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SAMPLE HANDLING AND ANALYSIS PLAN

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FOREWORD

This report is submitted by Space Operations/Integration and Satellite Systems Division of Rockwell International to the National Aeronautics and Space Administration's George C. Marshall Space Flight Center (MSFC) in accordance with contract NAS8-32953. Prepared by R. A. Happe, the Principal Investigator and K. S. Kim, the report covers the results of the investigation, "Containerless Preparation of Advanced Optical Glasses" performed for the period from April 12, 1978 to February 28, 1982.

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SUMMARY

The Containerless Processing of Advanced Optical Glasses Project, NAS8-32953, has been conducted in preparation for Space Shuttle MEA flight experiments. The investigation consisted of three technical tasks, a ground-based investigation, experiment/hardware coordination activities and development of flight experiment and sample characterization plans.

In the ground-based investigation over 100 potential candidate glass materials for space processing have been screened and promising compositions have been identified. In particular, the system of Nb_2O_5 - TiO_2 - CaO has been found to be very rich with new containerless glass compositions and an extensive number of the oxides combinations has been tried resulting in a glass formation ternary phase diagram. The frequent occurrence of glass formation by containerless processing among the compositions for which no glass formation had been previously reported indicated the possibility and an advantage of containerless processing in a terrestrial environment.

A number of meetings and numerous discussions were held with the experiment hardware developers to exchange information and to acquaint them with the hardware requirements. The Acoustic Chamber Positioning Module (ACPM) developed by Jet Propulsion Laboratory (JPL) and the Single Axis Acoustic Levitator (SAAL) by Interasonics were each evaluated for the applicability to the particular flight experiment. Following the selection of the hardware developer, guidance and technical information were provided to them, and assistance was provided to NASA in their review of the contractor's work.

As for the experimental plans, a detailed Experiment Requirements and Implementation Plan (ERIP) (an original plus a revision) has been prepared

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describing the experiment samples, experimental procedures and sequences, etc. A detailed Sample Handling and Analysis Plan (SHAP) has also been prepared describing the characterization methods and procedures for the flight experiment samples as well as the ground-prepared samples. The usefulness of these plans extends beyond the particular flight experiment and samples for which they were prepared, for they can be easily modified to meet the changing needs of many future flight experiments.

The major portion of this report has previously been reported in various forms to meet the reporting and documentation requirements during the project period. The underlying theme of this report is to present a coherent picture of the study accomplishments by weaving various reports together. Monthly progress reports, ERIP and SHAP all contained the results of the investigation. Especially, the information presented at the experiment review meetings is invaluable to understand the progress, issues, problems and future plans of the investigation at the time and presents a clear sense of an investigative process. For this reason the entire set of briefing charts presented at the three experiment review meetings are reproduced as Appendix A.



INTRODUCTION

If the promise of containerless melting and cooling, made possible by space processing, is realized fully in the years that lie ahead, an important new area of optical glasses will become a reality. In part, this new area may be visualized by referring to the schematic of Figure 1. The ordinate is the index of refraction, and the abscissa, the Abbe number (v), an inverse measure of dispersion. The higher Abbe numbers, to the left, of the diagram indicate a low dispersion (i.e., a flatter slope of the index versus wavelength curve). The lower Abbe numbers, to the right, have a high dispersion (steep index versus wavelength curve). A century ago flint glasses were developed. This permitted construction of the first achromatic, or color-corrected, multi-element lenses. Responding to the demands for better quality lenses, the optical glass industry developed more glasses with properties between those of the crown and flint glasses. More recently glasses have been developed to fill out the vertically hatched commercial glass area. The trend has been to push the area up and to the left with glasses of complex compositions.

If glasses beyond the reach of current terrestrial technology could be prepared from the more reluctant glass forming oxides, the area of useful properties could be expanded significantly. The expansion would occur by the addition of space-prepared glasses (horizontally hatched area of Figure 1) to the terrestrial base.

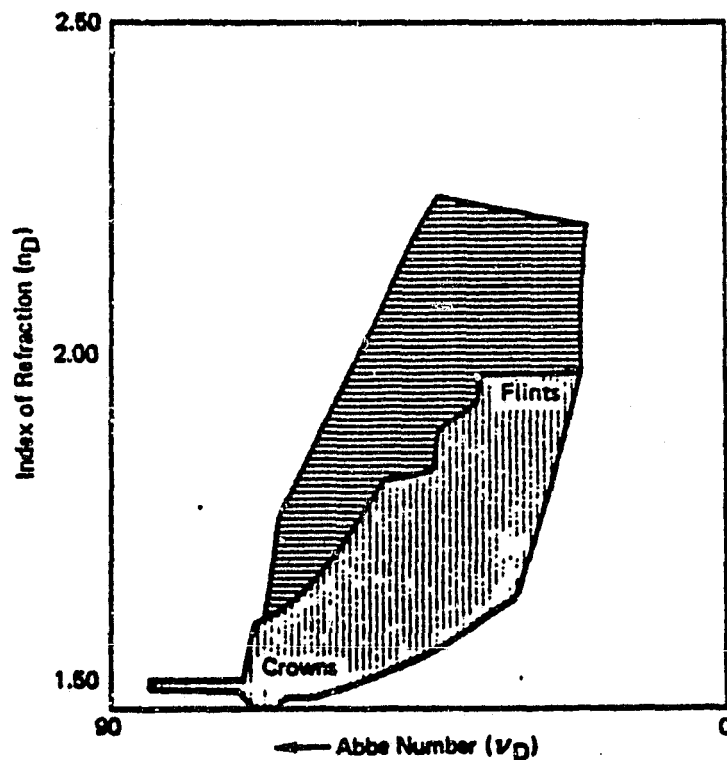


Figure 1. n-v Diagram for Optical Glasses

THE RATIONALE FOR SPACE PROCESSING

For most of the past decade, the Principal Investigator investigated possibilities for producing new optical glasses by containerless melting and cooling utilizing the near-zero-gravity environment available in earth orbit. The paragraphs that follow cover the technical thinking behind the concept of containerless processing in space, a summary of experience to date, and reasons for the interest in space processing of optical glasses.

When a molten oxide is cooled slowly enough to approach equilibrium conditions, it crystallizes near its crystalline melting point. In the case of the conventional glasses, usually based on oxides such as SiO_2 , P_2O_3 , or GeO_2 , the viscosity of the molten glass is very high. With this high

viscosity, the molecular mobility is very low. Thus, when such substances are cooled from the molten state, it is difficult for the molecules to rearrange themselves into the orderly state of the crystalline lattice. Because of this sluggishness, the movement of the molecules into the crystalline lattice positions is incomplete on cooling with normal cooling rates. In these cases, the semi-random molecular arrangement of the liquid state is essentially preserved on cooling, and the substance remains amorphous, the resulting product being called a glass.

The crystallization phenomenon may be considered to occur in two stages: (1) nucleation and (2) crystal growth. In conventional glasses the sluggishness effectively inhibits both of these processes, especially the latter. Therefore, even if the substance manages to nucleate on cooling from the melt, the crystal growth rate is so slow that the nuclei remain, for practical purposes, undetectable in the glass. There are, however, only a few oxides that have sufficiently high viscosities to permit glass formation under normal circumstances.

For the past two decades, glasses have been made terrestrially in the laboratory from some of the less viscous oxides. Invariably, the technique used for preparing them involves extremely high cooling rates from the liquid state. The familiar splat-cooling technique (Reference 1) is a case in point. While such techniques yield valuable research information about the nature of the glasses so prepared, their application for commercial purposes is extremely limited. By the nature of the technique, only very thin films can be prepared. With this technique the liquid, as a very thin layer, is cooled

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in contact with a chill plate, usually of copper. While the copper provides numerous nucleation sites, the very rapid cooling effectively suppresses crystal growth.

Containerless melting in space offers the first practical opportunity to prepare glasses in massive form from the large number of oxides whose liquid viscosity* is not high. If nucleation can be prevented on cooling, then crystal growth obviously cannot occur, and a glass should result.

* It is recognized that the slope of the viscosity versus temperature curve below the crystalline melting point (i.e., in the supercooled region) is very important to the glass formation process. However no such data exist for the oxides proposed here. It is probable that the general tendencies of viscosity change in the supercooled region can be inferred from future terrestrial and space studies.

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It is generally recognized that there are two kinds of nucleation: (1) heterogeneous and (2) homogeneous. Heterogeneous nucleation results from contact of the cooling liquid with crystalline material. Such a material may be entirely different in chemical composition from the melt. Common container wall materials are cases in point. Of course, it can also be of the same or similar composition, for example, unmelted portions of the bath or cool seed crystals of similar composition deliberately introduced into the cooling melt. In practice it is very difficult, or virtually impossible, to eliminate heterogeneous nucleation sites with conventional, terrestrial practice. Normally, a crystalline container must be used both for melting and for cooling. Further, the impingement of cool dust particles on the cooling melt may be enough to cause heterogeneous nucleation, and if the viscosity remains low enough in the supercooled liquid, crystal growth rates will be high and the glassy state will not be obtained.**

** The presence of insoluble crystalline material in the melt could also cause heterogeneous nucleation. Fortunately oxides are very good solvents. It therefore follows that with enough melting time this problem should be held to a minimum.

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Homogeneous nucleation is another matter. Theoretical studies (Reference 2) have shown that homogeneous nucleation rates for oxide glasses are much slower than for heterogeneous nucleation. Experimentally, it is difficult to determine whether nucleation is truly homogeneous. There are those who believe that it may never truly have been observed in an oxide glass. Since only a few molecules of a heterogeneous nucleator need to be present, the detection of such a small amount is a formidable technical problem. Thus, the assumption, *a priori*, that nucleation which occurs, for example, throughout the mass of a cooling substance is homogeneous may be erroneous. One can always argue that an undetectably small amount of a crystalline substance was present at the nucleation sites. At any rate, if heterogeneous nucleation can be effectively prevented, it is probable that homogeneous nucleation, if it can occur, will not occur unless the cooling rate is quite slow.

Over the past several years, the Principal Investigator successfully prepared numerous approximately 6-mm diameter (about 3/4 gram) glass boules of roughly spherical shape from several oxide compositions that have low viscosity in the molten state. That work is covered in detail in References 3 and 4. Among the compositions prepared are the gallia-calcia eutectic at approximately 19 weight percent calcia, an alumina-calcia composition with 30 weight percent calcia, and a ternary, 40 weight percent lanthana-40 weight percent alumina-20 weight percent calcia composition. The alumina-calcia composition is well outside the reported glass-forming region based on 20 mg melts (Reference 5). The gallia-calcia composition had been reported to be a glass former in the laboratory, but in sizes less than 40 mg (Reference 6).

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Furthermore, water quenching was required to achieve the glassy condition. Thus the preparation of crack-free boules with 50 times the mass of those of the earlier work represents a significant technical achievement. A glass of the ternary composition, to the Principal Investigator's knowledge, had never been reported in the literature.

The method for preparing the 6-mm boules is described in detail in Reference 3. Briefly, the samples in contact with a silica (glass) sting are suspended in a vertical air column. The energy for melting comes from a CO_2 laser beam aimed at one side of the boule. The silica sting was found necessary to stabilize the motion of the melt and is a definite convenience for getting the process started. The oxide is transferred to the sting from a laser melted area of well-mixed powders of the desired composition. While the technique developed by the Principal Investigator is excellent for demonstrating that new glasses can indeed be prepared with containerless melting and cooling techniques, it does suffer from several limitations, as follows:

1. Because of the relationship among viscosity, surface tension, and mass, 6 mm is very near the maximum sized boule that can be prepared in this fashion.
2. The silica sting material continuously dissolves into the sample during melting and holding at superheat temperature. Simultaneously, the sample constituents as well as silica are boiled out of the "hot spot" where the laser beam impinges. The end result is a net increase in the silica content. In order to keep the silica content as low as possible it is necessary to keep the melting time as short

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as possible (on the order of 30 seconds). As a consequence then, good mixing is not obtained.

3. It is very difficult to eliminate dust in the air from the wind tunnel. Thus the molten sample can be considered to be continually bombarded by dust particles while it is cooling. For this reason the technique may be unnecessarily restrictive compared with the more favorable conditions expected to accrue from space melting.

Space melting promises to eliminate or significantly reduce all three of the shortcomings inherent in the air suspension/laser melting equipment. Very large boules should ultimately be possible if enough power for melting can be made available. The silica sting will not be required with the acoustic positioning technique being developed by NASA. Since space melting can be accomplished in a furnace with nearly isothermal conditions, the localized heating of the specimen intrinsic in the terrestrial laser melting technique will not be experienced. Space melting can be accomplished in an essentially static atmosphere, significantly reducing the possibilities for dust-caused nucleation. It is entirely possible that some of the compositions that failed to form glass in our terrestrial experiments may prove to be glass formers under space melting and cooling conditions.

THE SOUNDING ROCKET PROGRAM

The Space Processing Applications Rocket (SPAR) program was considered a precursor to the Shuttle and later manned orbital programs. While conditions were not ideal for glass melting aboard a sounding rocket, largely because of the rather short melting time (less than five minutes) available, the program

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did afford a good opportunity to gain early experience with glass melting within the limitations and, more importantly, with space glass melting equipment development.

The gallia-calcia composition mentioned in the previous section of this report was originally chosen as a suitable composition for two sounding rocket experiments and for early Shuttle experiments using the NASA-provided Materials Experiment Assembly (MEA). The composition is a eutectic between the compounds $\text{CaO}:\text{Ga}_2\text{O}_3$ and $\text{CaO}:2\text{Ga}_2\text{O}_3$ and its composition, under equilibrium conditions, is approximately 19 wt. % CaO , balance Ga_2O_3 (approximately 56 mol % CaO). The phase diagram for the binary gallia-calcia system is shown in Figure 2.

This particular composition was chosen for the following reasons:

1. It has the lowest melting temperature of any of the new optical glass compositions studied by the Principal Investigator prior to the initiation of the SPAR program.
2. It is a relatively good glass former in the $\frac{1}{4}$ -inch (approximately 6mm) (about 800 mg) size under terrestrial containerless melting conditions.
3. It potentially has optical properties of interest to the optics industry.
4. Prior to our terrestrial melting work, it had not yet been prepared in a size exceeding 50 mg.

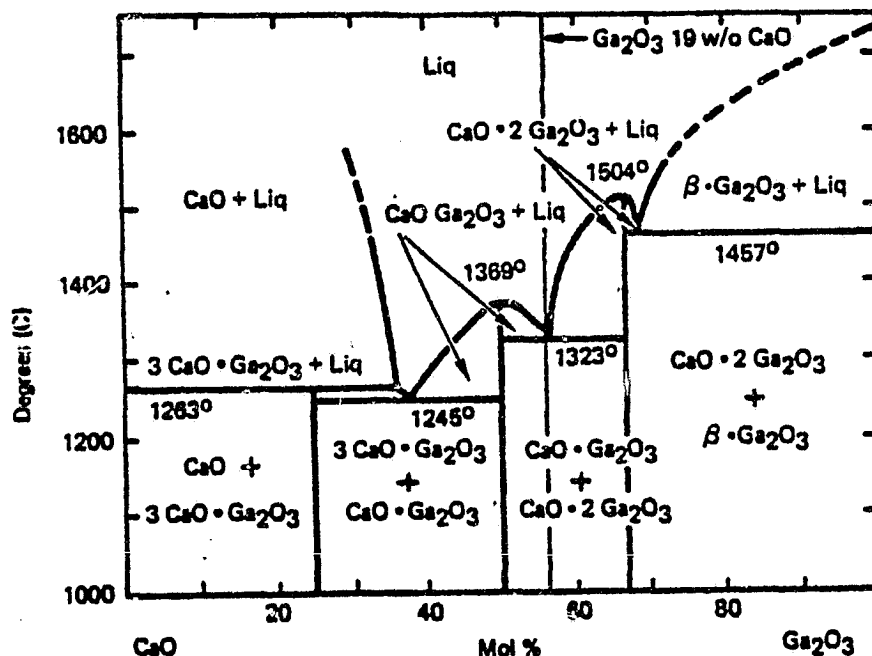


Figure 2. The $\text{CaO} - \text{Ga}_2\text{O}_3$ Phase Diagram (From Reference 7)

Despite these advantages and uniqueness of the binary composition, both SPAR VI and VIII glass experiments carried a silica-modified gallia-calcia composition. The ternary 39.3 Ga_2O_3 :35.7 CaO :25.0 SiO_2 (in mol percent) composition was chosen for the reason that this would provide more information about the function of the flight experiment hardware than the binary. Namely, in case of an incomplete melting, partial crystallization is expected of the ternary because of its slow crystal growth rate, whereas complete crystallization is expected of the binary composition. The results of the SPAR VI and VIII experiments have been reported in References 8 and 9, respectively.

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THE SHUTTLE MEA PROGRAM

As a part of the NASA Materials Processing in Space (MPS) program, the Shuttle MEA program is intended to provide an opportunity to conduct preliminary MPS experiments before they can be scheduled for the operational Shuttle/Spacelab missions. Its initial goal is to demonstrate the value of space for materials work by achieving significant scientific results and/or developing specific useful materials and products. This program utilizes a self-contained experiments system called Materials Experiment Assembly (MEA) which Marshall Space Flight Center is developing. The MEA consists of systems and apparatus developed for the Space Processing Applications Rocket (SPAR) project and other projects. The sounding rocket's most serious limitation is the short, low gravity time available. While every attempt is being made to prepare crystalline starting slugs of uniform composition on a macro scale, micro inhomogeneities must exist because of the multiphase nature of the composition. Therefore it is possible that some striae may be found in the rocket melted specimens because not enough time is available for complete mixing to take place by diffusion, the only significant mixing process predicted under near zero gravity conditions. In the Shuttle, by contrast, much longer melting times are possible, virtually assuring an opportunity to achieve homogeneous melts by diffusion. The longer melting time also permits larger boules to be melted as well as makes possible the performance of many experiments per mission.

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OBJECTIVES AND TASKS OF CONTAINERLESS PREPARATION OF ADVANCED OPTICAL GLASS PROJECT

The ultimate objective of this project was to pave the way for the Shuttle MEA experiments to develop new or unique optical glasses by processing marginal glass formers in a containerless low-gravity environment. The specific objectives were to select, by ground-based containerless processing, promising glass compositions that will be further investigated in the Shuttle experiments, to coordinate the experiment/hardware development activities and to develop the experimental plans.

The technical tasks included:

- (1) Conduct preparatory ground-based research program leading to the selection of the candidate materials to be processed during the MEA flight.
- (2) Perform experiment/hardware coordination activities.
- (3) Prepare an Experiment Requirements and Implementation Plan (ERIP) and a Sample Handling and Analysis Plan (SHAP).
- (4) Prepare reports on experimental progress and findings.

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GROUND-BASED INVESTIGATION

The purpose of this task was to select and understand the glasses and processing variables to be used in the flight experiment. This required performing an initial screening test on candidate materials with melting points below 1600°C.

APPARATUS AND TECHNIQUES

The main features of the apparatus and the techniques used in the ground-based experiments were the same as those used for the SPAR and an earlier program (Refs. 3 and 4). Namely, the laser melting/air suspension technique using a wind tunnel, a laser, and a sting was employed. The basic set-up is shown in Figs. 3 and 4. A compacted composition melted by a high power laser was suspended by a sting and further supported by a column of air from a vertical wind tunnel. To promote homogeneous melting the sting was rotated. The following steps highlight the experimental technique:

- (i) Screen powders (50 mesh)
- (ii) Weigh charges
- (iii) Mix-tumble (6 hours)
- (iv) Cold press into pellets (if possible)
- (v) Laser melt on copper (or aluminum or steel) plate
- (vi) Laser melt on sting with air suspension.

The laser used in the experiment was a cw CO₂ laser (10.6 μm) with an approximate output power of 1.5 kw. A raw beam of 9 cm-diameter was manipulated by the use of a series of optical components (mirrors, lenses and beam-splitter) to a narrow, slowly converging beam with an approximate diameter of 1 cm at the intersection of the beam and the sample.

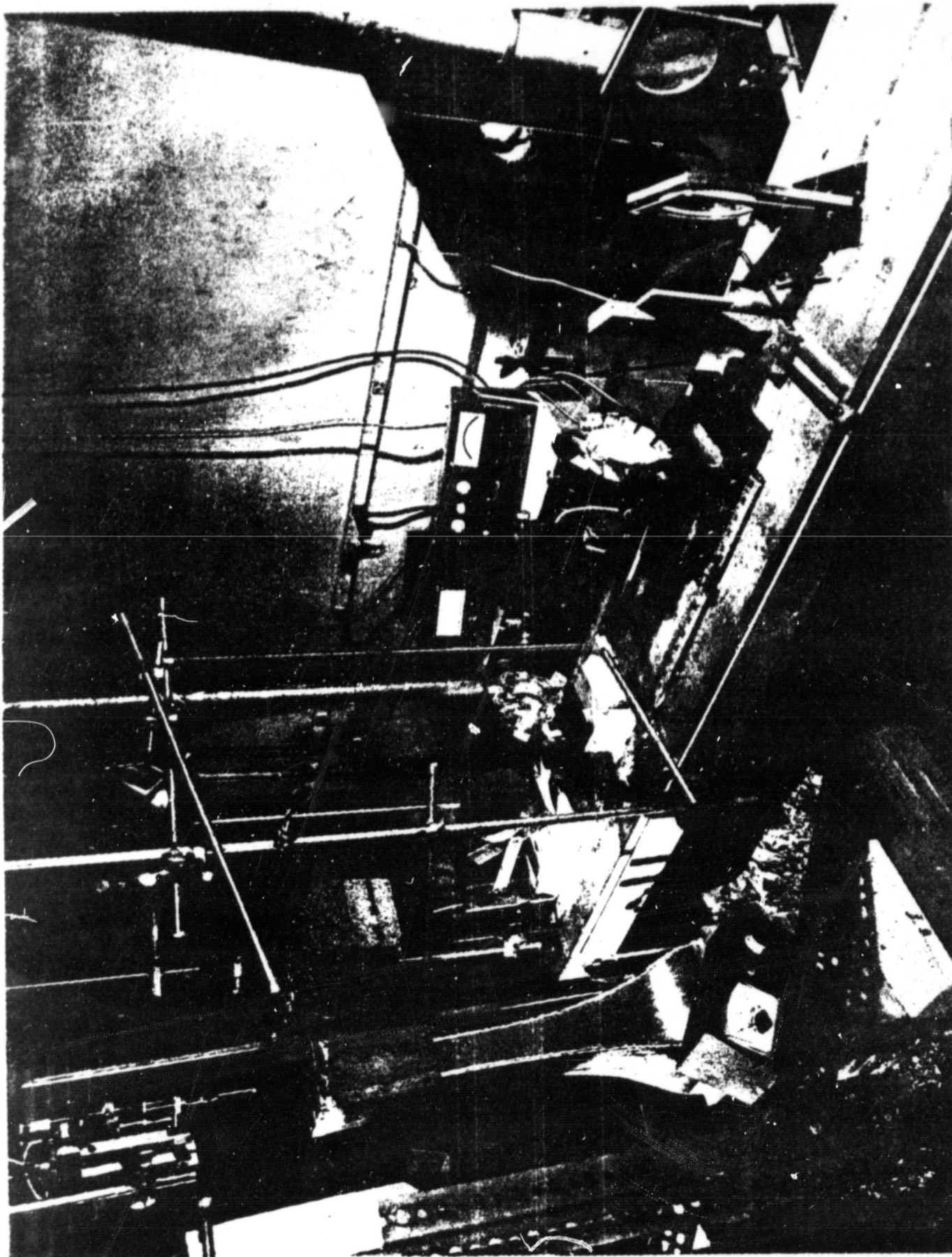


Figure 3. Optical Bench for Focussing and Directing Laser Beam

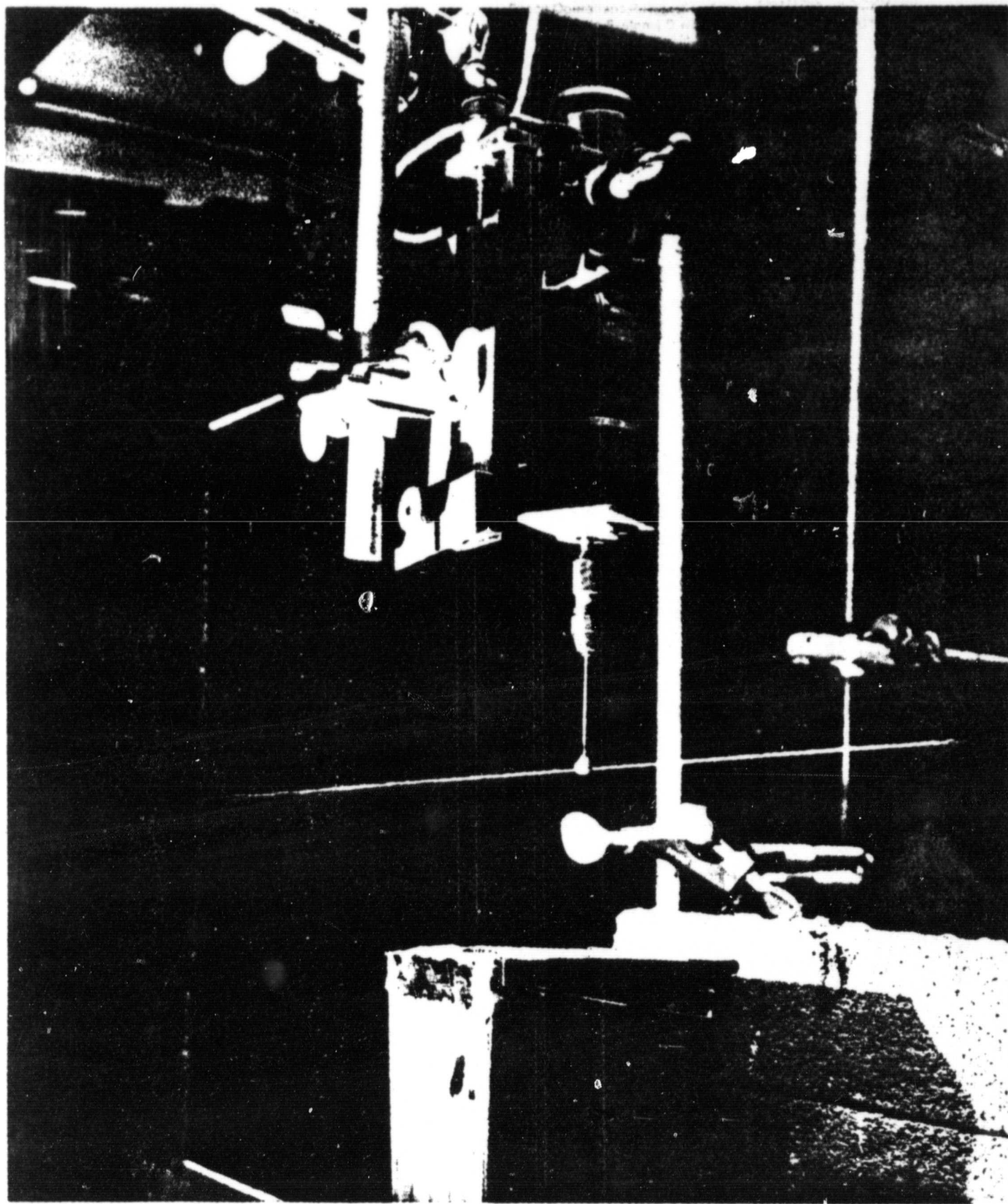


Figure 4. Sample Sting and Laser Beam

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A silica sting with a candidate composition fused to one of its ends was chucked in a vertical direction on a slow speed motor. As the specimen became molten on the slowly rotating sting, it was further supported by a column of air from the wind tunnel.

The vertical wind tunnel was specially designed for the purpose of supporting melts on the sting in the laser melt experiments. A detailed description of the wind tunnel may be found in Ref. 3.

RESULTS

A total of 105 new oxide compositions were tested for glass formation using the laser melting/air suspension technique. A great majority of these compositions, namely 83, had been originally listed in the Master List of Oxide Compositions (prepared in November 1978) as initial candidate compositions. This list was compiled from the oxide compositions that were mostly eutectic, had melting points below 1600°C , and mostly had not been previously reported for glass formation. In the course of the experiments an auspicious tendency for glass formation was discovered for combinations of Nb_2O_5 , TiO_2 , and CaO . A series of combinations of these oxides were tested in time making up the rest of the 105 compositions. Table 1 shows the results of the tests on all the 105 compositions. The results for the system of Nb_2O_5 - TiO_2 - CaO are summarized in their ternary phase diagram (Fig. 5) with the tentative containerless glass forming region indicated by the hatched area. The portion of this region whose melting points are 1500°C or lower is shown in Fig. 6 again by the hatched area.

TABLE 1 RESULTS OF LASER/AIR SUSPENSION SCREENING TESTS

MASTER LIST NO.	OXIDE CONTENT* WT. % (MOL %)	M.P., °C	OBSERVATIONS AND REMARKS
M1.1	77Nb-23Ca (41.4:58.6)	1488	Glass
M1.2	94.7Nb-5.3Sr (87.5:12.5)	1300	Crystal (Black)
M1.3	91.5Nb-8.5Ti (76.4:23.6)	1470	Deleted-see higher Ti content compositions which follow
M1.4	88Nb-12Ti (64:31)	1467	Crystal (Black)
M1.5	81Nb-19Ti (56:44)	1464	Crystal, SXGR**
M1.6	74Nb-26Ti (46:54)	1477	Crystal, RXGR*** (Same as 1.51)
M1.7	64.4SrNb ₂ O ₆ -32.6BaNb ₂ O ₆ (12:88)	1437	Crystal SXGR (Grey) (Made from carbonates)
M1.8	81.5Nb-16.7Ba-1.8Sr (71:25:4)	1305	Crystal SXGR
M1.9	71Nb-3.6Ba-25.3Sr (91SrNb ₂ O ₆ :9BaNb ₂ O ₆)	1465	Crystal, SXGR (Grey) (Made from carbonates)
M1.10	63.4Nb-16.3Sr-20.3Ba (45:30:25)	1430	Glass (Dk. Amber)

* Oxides listed by metallic element only
Corresponding oxides:

Nb₂O₅ SrO
TiO₂ BaO
CaO

** SXGR = Slow crystal growth rate, i.e., motion of the crystal growth front can be detected with the unaided eye in real time (total traverse time $\sim 1/30$ sec.)

*** RXGR = Rapid crystal growth rate-too rapid for motion to be detected.

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TABLE 1 RESULTS OF LASER/AIR SUSPENSION SCREENING TESTS (CON'T.)

MASTER LIST NO.	OXIDE CONTENT* WT. % (MOL %)	M.P., °C	OBSERVATIONS AND REMARKS
M1.11	74.4Nb-25.6Bn(62.5:37.5)	1320	Glass
M1.12	86Nb-14Zr(74:26)	1440	Crystal, SXGR
M1.13	23.6Nb-26.4Pb(70:30)	1334	Crystal
M1.14	63.2Nb-36.8Pb(59:41)	1310	Crystal, RXGR***
M1.15	39.2Nb-60.8Pb(35:65)	1225	Crystal, RXGR
M1.16	83.1Nb-16.9Zn(60:40)	1365	Crystal, RXGR
M1.17	60.6Nb-39.4Zn(32:68)	1285	Crystal, RXGR
M1.18	45Nb-55Zn(20:80)	1300	Crystal, SXGR**
M1.19	88Nb-12Na(63:37)	1220	Crystal, SXGR-Many nuclei (brownish cream color)
M1.20	84.6Nb-15.4K(66:34)	1150	Crystal, SXGR (Off White)
M1.21	49.3Nb-50.7Bi(63:37)	1180	Crystal, RXGR-Heavy vaporization
M1.22	78.3Nb-18.3Ni-2.26Ba-1.18Ti (60BaTiO ₃ :40NaNbO ₃)	~1330	Glass (lt. Yellow) (Xtal is black)- Fairly heavy vap.::

* Corresponding oxides:

Nb ₂ O ₅	ZnO
BaO	Na ₂ O
ZrO ₂	K ₂ O
PbO	Bi ₂ O ₃

** See Page 1 footnote

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TABLE 1 RESULTS OF LASER/AIR SUSPENSION SCREENING TESTS (CON'T.)

MASTER LIST NO.	OXIDE CONTENT* WT. % (MOL. %)	M.P., °C	OBSERVATIONS AND REMARKS
M1.23	93.7Nb-6.3Al(85:15)	1380	** Crystal-SXGR (Mottled grey "dk & lt") Single front growth
M1.24	98Nb-2Al(95:5)	1410	Crystal-Fairly SXGR (Mottled grey)
M1.25	66Nb-34Ca(29:71)	1590	Glass
M1.26	84.4Nb-15.6La(87:13)	1330	Crystal-SXGR (Mottled dk & lt. yellow)
M1.27	78.9Nb-21.1La(82:18)	1310	Crystal-Very SXGR (Mottled dk. & lt. yellow)
M1.28	63.5Nb-36.5La(68:32)	1350	Crystal and Gl. + crystal? Marginal glass former
M1.29	51Nb-49Ba(37.5:62.5)	1288	Glass (good glass former) (lt. amber)
M1.30	78.6Nb-21.4Cd(64:36)	1360	Deleted-Cd volatile. + toxic
M1.31	94Nb-6Li(64:36)	1200	Glass (clear with lt. yel. waste) Devitrifies on probe. Can get either clear (100%) glass, opaque, or translucent glass
M1.32	70.8Nb-21.7Ti-7.5Ca(39.7:40.4:19.9)	1350	Crystal SXGR (Black)
M1.33	79.9Nb-16.8Ti-8.3Ca(44.0:32.8:23.1)	1348	Crystal VSXGR (Black)
M1.34	80.7Nb-12.4Ti-6.9Ca(52.2:26.7:21.1)	1345	Crystal SXGR (dk. grey)

** See Page 1 footnote

* Corresponding oxides:

Nb ₂ O ₅	La ₂ O ₃	Li ₂ O
Al ₂ O ₃	BaO	Ti ₂ O ₃
CaO	CdO	

TABLE 1 RESULTS OF LASER/AIR SUSPENSION SCREENING TESTS (CON'T.)

MASTER LIST NO.	OXIDE CONTENT* WT. % (MOL %)	M.P., °C	OBSERVATIONS AND REMARKS
M1.35	84.5Nb-8.8Ti-6.7Ca(58.1:20.1:21.8)	1346	Crystal, SXGR (Dirty green color)
M1.36	90.9Nb-3.9Ti-5.2Ca(70.7:10.1:19.2)	1348	Crystal, SXGR (Dirty green color)
M1.37	52.5Nb-32.0Ti-15.5Ca(22.6:45.8:31.6)	1357	Crystal, VSXGR: (dk. grey-black)
M1.38	39.2Nb-43.8Ti-17.0Ca(14.8:54.9:30.3)	1395	Crystal, RXGR - Very mountainous
M1.39	57.5Nb-24.0Ti-18.5Ca(25.5:35.5:39.0)	1404	Glass (dk. brown)
M1.40	64.0Nb-16.3Ti-19.7Ca(30.2:25.6:44.1)	1435	Glass + small amount surface crystal (dk. amber)
M1.41	68.3Nb-8.7Ti-23.0Ca(33.1:14.0:52.8)	1500	Glass (dk. amber)
M1.42	65Nb-10Ti-25Ca(30.0:15.3:54.7)	1500	Crystal + 1/3 glass (all at bottom)
M1.43	60.5Nb-17.0Ti-22.5Ca(27.0:25.3:47.7)	1500	Crystal, SXGR (White) (Very exothermic)
M1.44	50Nb-28Ti-22Ca(20.2:37.6:42.1)	1500	Glass (Amber)
M1.45	57Nb-21Ti-22Ca(24.6:30.2:45.1)	1500	Glass

* Corresponding oxides:

Nb₂O₅, TiO₂, CaO

** See footnote, Page 1

*** See footnote, Page 1

TABLE 1 RESULTS OF LASER/AIR SUSPENSION SCREENING TESTS (CON'T.)

MASTER LIST NO.	OXIDE CONTENT* WT. % (MOL %)	M.P., °C	OBSERVATIONS AND REMARKS
M1.46	79Nb-21Ca(44.7:55.8)	1500	Glass
M1.47	84Nb-16Ca(52.6:47.4)	1550	Glass (Congr. melt. compound)
M1.48	43.5Nb-31.5Ti-25.0Ca(16.3:39.3:44.4)	1650	Glass
M1.49	55Nb-14Ti-31Ca(22.1:18.7:59.1)	1650	Glass (lt.amber)
M1.50	43Ti-57Ca	1740	Glass + crystal (dk. colored)-Reluctant gl. former
M1.51	74Nb-26Ti	1477	Crystal Mod. RXGR***
M1.52	19.8Nb-31.8Ti-48.4Ca	1740	Glass (lt. amber)
M1.53	26.0Nb-25.4Ti-48.6Ca	1728	Glass (lt. amber)

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*** See footnote, Page 1

* Corresponding oxides:

Nb₂O₅ CaO
TiO₂

TABLE 1 RESULTS OF LASER/AIR SUSPENSION SCREENING TESTS (CON'T.)

MASTER LIST NO.	OXIDE CONTENT* WT. % (MOL %)	M.P., °C	OBSERVATIONS AND REMARKS
M2.1	97.8Ta-2.2Mg(80:20)	1600	Crystal-glossy (crystal + glass ?)
M2.2	87.6Ta-12.4K(60:40)	1550	Crystal, RXGR-difficult to suspend (Low surface tension + high density + low viscosity ??)
M2.3	KTaO ₃		Crystal, RXGR(White) (black xtal on Cu Plate)
M2.4	50Ta-50La	High	Deleted (covered by Topol-Happe in previous work)
M2.5	75Ta-25La	High	Deleted (covered by Topol-Happe in previous work)
M2.6	40(2CaO·Ta ₂ O ₅) + 60(CaO·SiO ₂)	1438	Deleted because of high SiO ₂ content
M2.7	96.6Ta-3.4Li(66:34)	1553	Glass-reluctant glass former (sl. bluish caste)
M2.8	68.9Ta-31.1K(32:68)	1090	Translucent glass on Cu plate, would not ball up-probably good gl. former-no further tests.

* Corresponding oxides:

Ta₂O₅, MgO, K₂O, La₂O₃, CaO, SiO₂, Li₂O

*** See Footnote, Page 1

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TABLE 1 RESULTS OF LASER/AIR SUSPENSION SCREENING TESTS (CON'T.)

MASTER LIST NO.	OXIDE CONTENT* WT. % (MOL %)	M.P., °C	OBSERVATIONS AND REMARKS
M3.1	36.5Al-63.5Nb(60:40)	1440	Crystal (grey) RXGR**
M3.2	10Al-10Ti-38Si-28Ca-14Mg (5.7:7.3:37.2:29.4:20.4)	~1250	Glass (black) on Cu mold plate-no air susp. tests
M3.3	65KAlSiO ₄ · 15SiO ₂ : 20Mg ₂ SiO ₄	1456	Deleted because of high SiO ₂ content (probably is conventional "container" glass)
M3.4	24Al-76Mn	1520	Glass ?? conchoidal fracture-looks crystal dull black surface, glossy at fracture- opaque
M3.5	60(39rO·Al ₂ O ₃)-40(3CaO·Al ₂ O ₃)	1580	Glass (dk. grey), (Good glass former)
M3.6	33Al-67Sr(33.3:66.7)	1505	Glass + crystal (formed translucent lt. blue- green glass (?) on Cu)
M3.7	62Al ₂ O ₃ -38BaO	1620	Crystal (white), Couldn't melt completely
M3.9	64Al ₂ O ₃ ·28CaO·8P ₂ O ₅	1590	Deleted because of high m.p. (probably containerless glass former)
M3.10	19.2Al-80.8W(35:65)	1230	Deleted-WO ₃ volatility
M3.11	23Al ₂ O ₃ ·77WO ₃	1350	Deleted-WO ₃ volatility

** See footnote, Page 1

* Corresponding oxides:

Al₂O₃, Nb₂O₅, SiO₂, CaO, MgO, MnO, SrO, WO₃

TABLE 1 RESULTS OF LASER/AIR SUSPENSION SCREENING TESTS (CON'T.)

MASTER LIST NO.	OXIDE CONTENT* WT. % (MOL. %)	M.P., °C	OBSERVATIONS AND REMARKS
M3.12	65(CaO·Al ₂ O ₃ ·2SiO ₂)- 35(CaO·TiO ₂)(47.6:52.4)	1350	Glass on Cu (black) No further testing
M3.13	73NaAlSiO ₄ ·27KAlSiO ₄	1484	Deleted because of high SiO ₂ content- probably conventional glass former
M3.17	94.5CaAl ₂ O ₄ -5.5MgAl ₂ O ₄	1593	Deleted-high melting point (similar to CaO·Al ₂ O ₃)
M3.18	(64Al ₂ O ₃ -36(Na ₂ O·Al ₂ O ₃))	1580	Deleted-high melting point (similar to CaO·Al ₂ O ₃)

* Corresponding oxides:

Al₂O₃, Na₂O, CaO, K₂O, TiO₂, MgO, SiO₂

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TABLE 1 RESULTS OF LASER/AIR SUSPENSION SCREENING TESTS (CON'T.)

MASTER LIST NO.	OXIDE CONTENT* WT. % (MOL %)	M.P., °C	OBSERVATIONS AND REMARKS
M4.1	78Ti-22Sc(86:14)	1560	Crystal-glossy (gl + xtal?)
M4.2	52.5Ti-47.5Ba(68:32)	1317	Glass (some arrested crystal growth)
M4.3	57.6Ti-42.4Zn(58:42)	1418	Crystal v. RXGR ^{***} (Quite exothermic)
M4.4	69.6Ti-30.4Na(64:36)	985	Crystal (lt. yellow) RXGR
M4.5	81.5Ti-18.5 Ca(75.5:24.5)	1460	Crystal SXGR ^{**} (Same results when made with CaTiO ₃)
M4.6	71Ti-29Sr(76:24)	1440	Glass (dk. amber) Reluctant glass former
M4.7	66.7Ti-33.3Mn(64:36)	1290	Crystal (brown) RXGR
M4.8	(86Ti:14Sc)	1560	Deleted (high m.p.)
M4.9	57Ti-43CaF ₂ (56.4:43.6)	1360	Glass + Crystal
M4.10	45(4TiO ₂ ·Al ₂ O ₃)-55CaF ₂ (34.4Ti:8.6Al:56.9CaF ₂)	1280	Glass (dk. amber)
M4.11	28TiO ₂ ·20(4TiO ₂ Al ₂ O ₃)-52CaF ₂ (43.0Ti:3.8Al:53.2CaF ₂)	1325	Glass (dk. yellow/grey) Good glass former
M4.12	54Ti-28Mg-16Al(46.3:43.5:10.2)	1570	Crystal SXGR (Lt. yellow)

* Corresponding oxides:

TiO₂, Sc₂O₃, BaO, ZnO, Na₂O, CaO, SrO,
MnO, Al₂O₃, MgO

** See Footnote, Page 1

*** See Footnote, Page 1

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TABLE 1 RESULTS OF LASER/AIR SUSPENSION SCREENING TESTS (CON'T.)

MASTER LIST NO.	OXIDE CONTENT* WT. % (MOL %)	M.P., °C	OBSERVATIONS AND REMARKS
M5.1	60Zr-40Na(43:57)	>1380	Crystal, Would not ball on Cu-high m.p.? Incompl. melting
M5.3	(27Zr:73W)	1231	Deleted high W ₀₃ -Volatile

* Corresponding oxides:

ZrO₂, Na₂O, W₀₃, SiO₂

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TABLE 1 RESULTS OF LASER/AIR SUSPENSION SCREENING TESTS (CON'T.)

MASTER LIST NO.	OXIDE CONTENT* WT. % (MOL %)	M.P., °C	OBSERVATIONS AND REMARKS
M6.1	66.7La-38Nb(62:38)	1590	Crystal, RXGR***
M6.2	82.9La-17.1Y(77:23)	1570	Glass + Crystal-difficult to melt completely
M6.3	68.7La-31.3Pb(60:40)	1130	Very volatile-splattered-test discontinued (some glass (?) on Cu)
M6.4	45La-55Ti(16.7:83.3)	1445	Glass (+crystal)(black) <u>Marginal gl. former</u>
M6.8	(20La:80W)	1570	Deleted-high m.p., high vol. WO_3

* Corresponding oxides: *** See footnote, Page 1

La_2O_3 , Nb_2O_5 , Y_2O_3 , PbO , TiO_2 , B_2O_3 , BaO , SnO , WO_3

TABLE 1 RESULTS OF LASER/AIR SUSPENSION SCREENING TESTS (CON'T.)

MASTER LIST NO.	OXIDE CONTENT* WT. % (MOL %)	M.P., °C	OBSERVATIONS AND REMARKS
M7.1	88.7Ga-11.3Ca(70:30)	1457	Glass (Colorless)
M7.2	67Ga-33Ca(38:62)	1245	Glass (Beer bottle amber)
M7.3	72Ga-28Sr(58.7:41.3)	1430	Glass (grey)
M7.4	44Ga-56Sr(30.3:69.7)	1250	Glass (v. dk. grey) (some gl. on Cu + on probe)
M7.5	78Ga-22Ti(60:40)	1500-1580	Crystal, RXGR***

* Corresponding oxides:
Ga₂O₃, CaO, SrO, TiO₂

*** See footnote, Page 1

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TABLE 1 RESULTS OF LASER/AIR SUSPENSION SCREENING TESTS (CON'T.)

MASTER LIST NO.	OXIDE CONTENT* WT. % (MOL %)	M.P., °C	OBSERVATIONS AND REMARKS
M8.1	100% PbO	875	Deleted-volatile
M8.2	71Zn-11Al-18Si(68.2:8.4:23.4)	1440	Heavy vaporization (glass on Cu)
M8.3	41Y-33Si-26Al(18.4:55.7:25.9)	~1400	Clear glass on Cu (paper ordered)
M8.4	PbMoO ₄	1065	Very heavy vaporization-no further testing
M8.5	Nd ₂ (WO ₄) ₃	1125	(Nd ₂ O ₃ ·3WO ₃)
M8.6	(66m/o Nd ₂ (MoO ₄) ₃ ·34m/oNd ₂ (WO ₄) ₃)	~1070	Deleted-volatility

* Corresponding oxides:

ZnO, Al₂O₃, SiO₂, Y₂O₃, Nd₂O₃, WO₃, MoO₃, V₂O₅, K₂O, TiO₂, Na₂O, TiO₂

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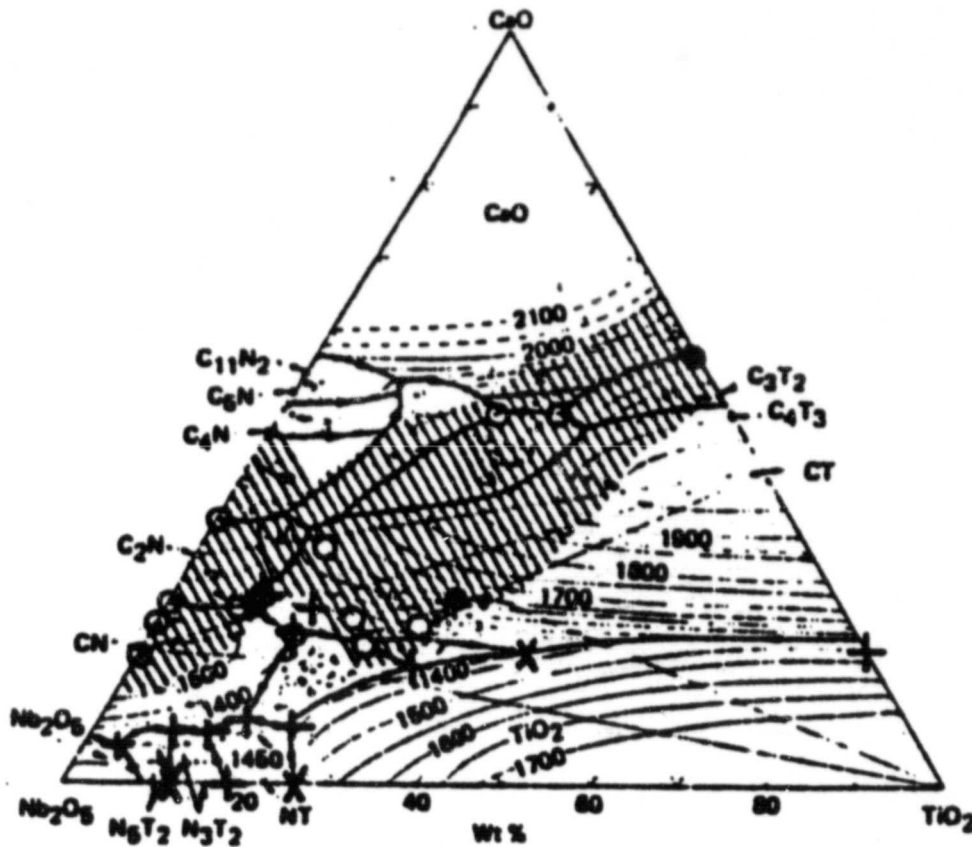


Fig. 5 Glass Formation Region in the Nb_2O_5 - TiO_2 - CaO System (Phase Diagram
in Ref.10)

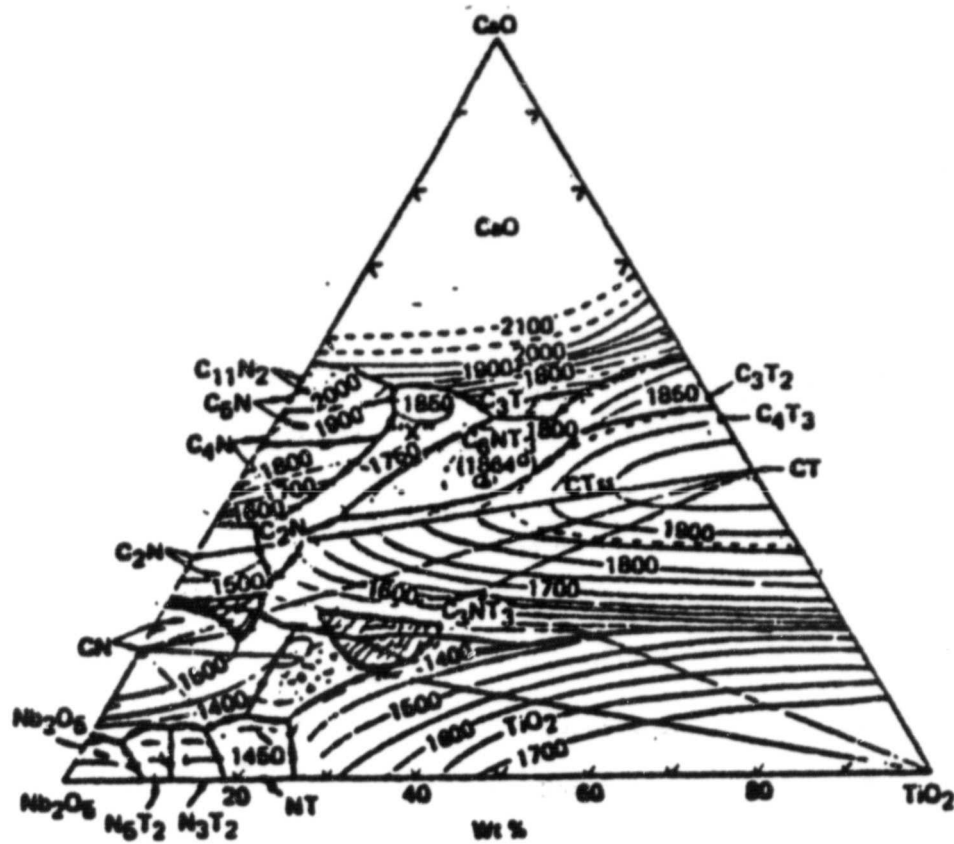


Fig. 6 Glass Formation Region in the Above System with $T_m < 1500^\circ\text{C}$

EXPERIMENT/HARDWARE COORDINATION ACTIVITIES

The experiment hardware to be used in an MPS experiment must work as an integrated system. Namely, the system must have integrated capabilities of heating, cooling, injecting/retrieving, positioning, photographing, etc., samples in addition to recording data during the experiment. The development/integration of such a system will be accomplished by a hardware developer under a contract from NASA, and the Principal Investigator is expected to provide guidance to the hardware contractor regarding the hardware requirements and to assist NASA in reviewing the adequacy of the contractor's work.

An essential feature of the experiment hardware for containerless processing in the low gravity environment is the levitation of a molten sample. Jet Propulsion Laboratory (JPL) developed ACPM (Acoustic Chamber Positioning Module) and Intersonics developed SAAL (Single Axis Acoustic Levitator) for this purpose, both under NASA contracts. The JPL's ACPM is a levitation chamber in which triaxial resonant acoustic waves are excited and a sample is levitated at the intersection of these waves. The Intersonics' SAAL is an acoustic levitator (requiring no chamber) in which a single vibrator excites an axial acoustic wave. The wave is reflected by a reflector and a sample is levitated at a node of the resulting standing wave.

Because of the different limitations imposed by these two levitators, an investigation was conducted to assess and compare their capabilities in terms of the experiment requirements. A number of meetings were held (4/78 at MSFC, 5/78 at Rockwell, 9/78 at MSFC, 10/78 at JPL, and 1/79 at MSFC) in which the feasibility of using the JPL's ACPM was discussed. Major issues

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raised in these discussions were the maximum temperature and the cooling rate capabilities vs requirements. Since the JPL's ACPM was not able to meet these requirements at the time it was decided in the early part of 1980 that the Interionics' SAAL be used in the flight experiments planned in the near future.

Many discussions were held with Interionics before and since then regarding the hardware requirements and modification, and a visit was made to them in May 1981 for an on-site discussion. Items discussed included the heating rate, maximum temperature, cooling rate, sample injection/retrieval mechanism, motion picture, levitator failure mechanisms, number and type of samples, etc.

FLIGHT EXPERIMENT PLAN AND SAMPLE CHARACTERIZATION PROCEDURE

Detailed requirements and plans were provided for the flight experiment and sample characterization. These were designed to help each discipline involved in the integration and the support of the experiment be familiar with their respective objectives and requirements, and also form a basis for discussion among them.

EXPERIMENT REQUIREMENTS AND IMPLEMENTATION PLAN (ERIP)

The purposes of this plan were: to describe the current status of the experiment and update information in the proposal; to update experiment requirements; and to provide program plans in the form of milestone schedules. Specifically, the plan included experiment definition, description of experiment specimens, prelaunch and flight operations, and flight and experiment data acquisition requirements. The initial plan prepared in June 1978 (SD78-AP-0071) was superseded by a revised plan in July 1981 (SD78-AP-0071 REV A). The revised plan in its entirety is reproduced in this report as Appendix B.

SAMPLE HANDLING AND ANALYSIS PLAN (SHAP)

The basic objective of this plan was to perform an analysis of the experiment samples that would help evaluate the accomplishments of the overall flight experiment. The plan was specifically developed to characterize and compare samples produced under ground-based and flight conditions. It included the methods and techniques to be used in analyzing the samples, handling procedures, and a sequence of the entire characterization process.

In addition to this basic plan, another plan (in a draft form) was developed in which an extensive set of measurements were tentatively proposed

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to fully characterize the experiment samples, ground-based and space-produced, to explore various application potentials. The basic SHAP prepared in June 1981 (SSD81-0119) and the draft plan (prepared in May 1981) are attached to this report as Appendices C and D, respectively.

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CONCLUSION

By uncovering many new containerless glass compositions, the screening experiments performed in this program has possibly opened the door to broad, new types of glass which could become a reality through space processing. Optical properties hitherto unobtainable (in terms of, e.g., refractive index, dispersion, and absorption properties) could be obtained and made use of in designing optical components such as lenses, filters and windows.

The frequent occurrence of new glass formation found in the present metallic oxide system suggests the possibility that new and unique glasses could also be found by containerless processing in other systems such as the halide and the chalcogenide systems.

In the present screening tests the melting points in most cases were kept below 1600°C . This temperature restriction imposed because of the flight hardware requirements was an arbitrary one as far as glass formation was concerned. In fact, preliminary glass formation was already observed in the temperature range above 1600°C . Potential for glass formation above 1600°C appears very good, and work in that region should be encouraged.

Writing in the Physic Today (November 1981) on the development of optics in the past fifty years (or rather 20 years), Peter Franken, the director of the Optical Science Center of the University of Arizona, cited three emergences as being most responsible for the recent dramatic advances in optics: laser, high-speed computer, and new materials. Space processing, when fully understood, serving as an additional material processing parameter possesses an immense value for development of new materials.

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ACKNOWLEDGEMENTS

The writer wishes to acknowledge that the Principal Investigator performed the entire ground-based investigation, conducted the major portion of the experiment/hardware coordination effort, and made significant contribution to the development of ERIP and SHAP.

The Principal Investigator and the writer wish to express their thanks to L. E. Topol of Rockwell for the valuable suggestions on the screening experiments, and D. Gnanamuthu and R. Moore of the Rockwell Science Center for their cooperation in the installation of the experimental apparatus.

Many valuable conversations with C. Ray of Intersonics on the experiment hardware and the administrative assistance rendered by R. Fallon of MSFC are also acknowledged.

Thoughtful guidance and advice given us throughout the project period by L. L. Bissing of Rockwell are gratefully acknowledged.

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MARCH 19, 1960

CONTAINERLESS PREPARATION OF
ADVANCED OPTICAL GLASSES - EXPERIMENT M770095

CONTRACT NAS8-32853

R.A. HAPPE, PRINCIPAL INVESTIGATOR
ROCKWELL INTERNATIONAL

OUTLINE -- SCIENCE REVIEW 3/19/80

I OBJECTIVES

- II RATIONALE FOR SPACE PROCESSING OF OPTICAL GLASSES**
- III INVESTIGATION PLAN**
- IV SCREENING RESULTS**
- V NEXT YEAR'S WORK**

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APPENDIX A

3

OBJECTIVES

- DEVELOP NEW OXIDE-BASED GLASSES WITH NEW AND USEFUL OPTICAL PROPERTIES USING CONTAINERLESS PROCESSING IN THE VERY LOW GRAVITY OF SPACE
- DEVELOP SCIENTIFIC INFORMATION NECESSARY TO ACHIEVE THE ABOVE
 - UNDERSTANDING OF
 - CRYSTAL NUCLEATION
 - CRYSTAL GROWTH
 - ROLE OF VISCOSITY
 - EFFECTS OF COMPOSITION ON PROPERTIES

APPENDIX A

OUTLINE - SCIENCE REVIEW 3/19/80

I OBJECTIVES

✓ II

RATIONALE FOR SPACE PROCESSING OF OPTICAL GLASSES

III INVESTIGATION PLAN

IV SCREENING RESULTS

V NEXT YEAR'S WORK

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CONVENTIONAL GLASSES

- SiO_2 , P_2O_5 , B_2O_3 , GeO_2
- VERY HIGH MOLTEN VISCOSITY
- STEEP SLOPE OF TEMPERATURE VS. VISCOSITY CURVE
- DIFFICULT FOR MOLECULES TO REARRANGE THEMSELVES INTO CRYSTALLINE ARRAY ON COOLING
- AMORPHOUS STATE IS PRESERVED - GLASS

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**CRYSTALLIZATION
FROM THE LIQUID STATE.**

- NUCLEATION
- CRYSTAL GROWTH

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GLASSES FROM LESS VISCOUS OXIDES

- PREVENTION OF CRYSTAL GROWTH
 - VERY RAPID COOLING (SPLAT)
 - VERY THIN FILMS ONLY
- AVOIDANCE OF NUCLEATION
 - CONTAINERLESS PROCESSING
 - MORE MASSIVE SHAPES

CRYSTAL NUCLEATION

- HETEROGENEOUS - STARTS FROM CONTACT WITH CRYSTALLINE MATERIAL
 - CONTAINER WALLS
 - UNDISSOLVED MATERIAL - SAME OR DIFFERENT COMPOSITION AS THE MELT
 - SEED CRYSTALS
 - DELIBERATELY INTRODUCED
 - ACCIDENTALLY INTRODUCED - I.E., DUST
- HOMOGENEOUS (SPONTANEOUS OR SELF-)
 - MUCH SLOWER RATES THAN HETEROGENEOUS
 - RARELY, IF EVER, OBSERVED - SLOW COOLING RATES

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TERRESTRIAL RESEARCH

- LASER SPIN MELTING

- LASER/AIR SUSPENSION MELTING AND COOLING

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TERRESTRIAL CONTAINERLESS PROCESSING LIMITATIONS

- 6 MM NEAR MAXIMUM SIZE
- CONTACT WITH SILICA STRING
- DUST MANAGEMENT

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LOW-GRAVITY CONTAINERLESS PROCESSING IN SPACE

- LARGER THAN 6 MM
- NO STING REQUIRED
- STATIC ATMOSPHERE - LESS, OR NO (7) DUST IMPINGEMENT WHILE COOLING

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OTHER ADVANTAGES OF CONTAINERLESS PROCESSING

- PERMITS MELTING OF HIGH MELTING TEMPERATURE MATERIALS, SOME OF WHICH MAY HAVE HIGH VISCOSITY NEAR CRYSTALLINE MELTING POINT
- PERMITS ADJUSTMENT OF ATMOSPHERE TO NEUTRAL TO PARTICULAR SUBSTANCE(S) BEING MELTED

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- I OBJECTIVES
- II RATIONALE FOR SPACE PROCESSING OF OPTICAL GLASSES
- ✓ III INVESTIGATION PLAN
- IV SCREENING RESULTS
- V NEXT YEAR'S WORK

INVESTIGATION PLAN -- PHASE II GROUND RESEARCH
NAS8-32953

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	% COMPLETE
1. SCREEN CANDIDATE OXIDE COMPOSITIONS	80
2. PROCEDURE DEVELOPMENT FOR FLIGHT SAMPLE PREPARATION	0
3. PREPARE AND CHARACTERIZE FLIGHT SAMPLES	0
4. FLIGHT EXPERIMENTS	0
5. POST-FLIGHT EVALUATION AND REPORT	0

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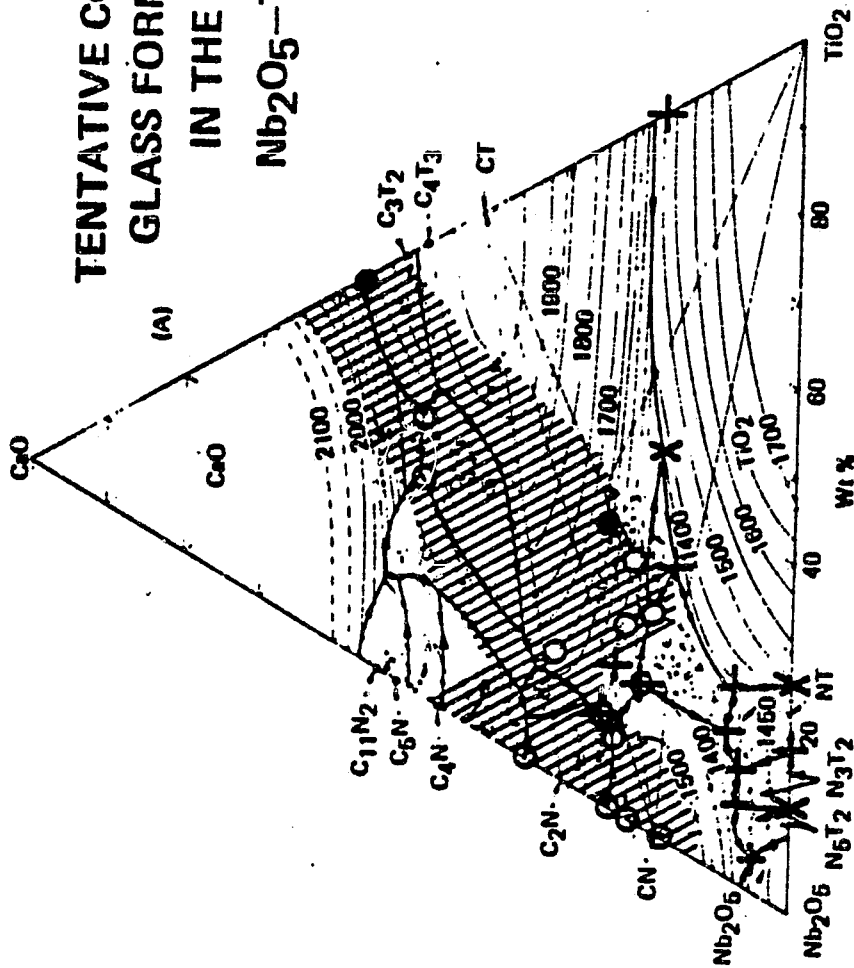
- I OBJECTIVES
- II RATIONALE FOR SPACE PROCESSING OF OPTICAL GLASSES
- III INVESTIGATION PLAN
- ✓ IV SCREENING RESULTS
- V NEXT YEAR'S WORK

SYMBOLS USED FOR LASER/AIR SUSPENSION SCREENING TEST RESULTS

- GLASS
- RELUCTANT GLASS FORMING COMPOSITION.
- ⊕ GLASS + CRYSTAL
- + CRYSTAL - SLOW CRYSTAL GROWTH RATE.
- x CRYSTAL - RAPID CRYSTAL GROWTH RATE.

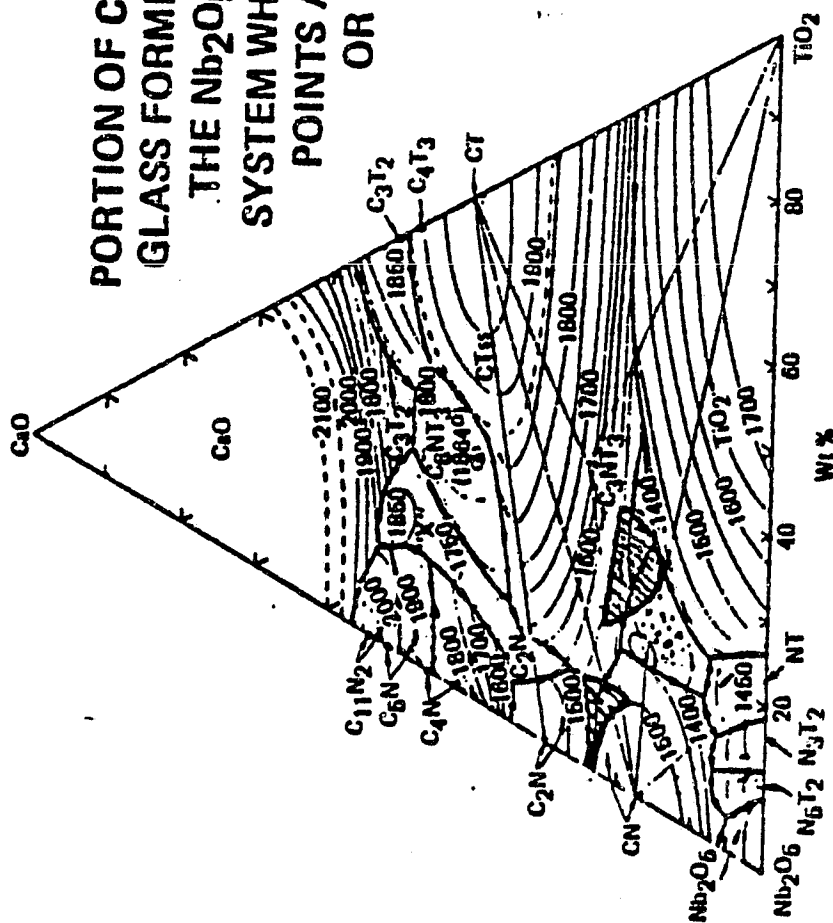
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TENTATIVE CONTAINERLESS
GLASS FORMING REGION
IN THE SYSTEM,
 $\text{Nb}_2\text{O}_5\text{--TiO}_2\text{--CaO}$

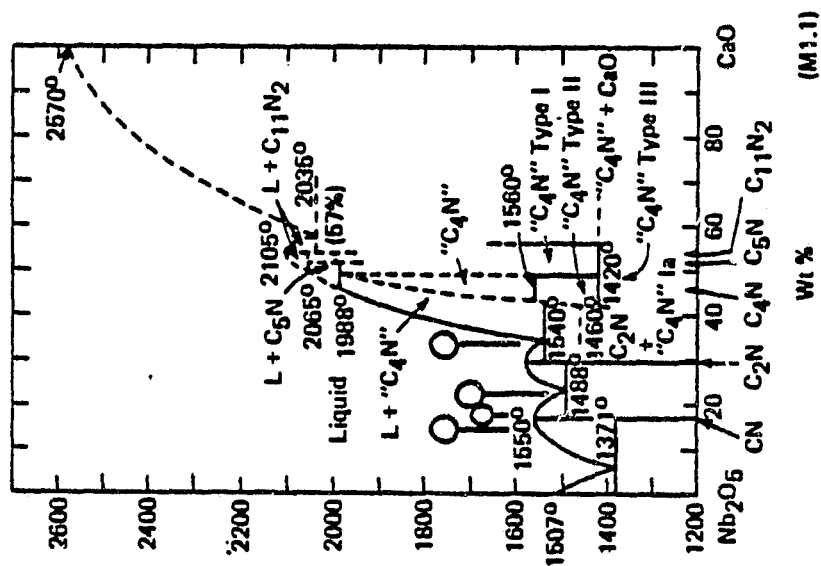


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PORTION OF CONTAINERLESS
GLASS FORMING REGION OF
THE Nb_2O_5 - TiO_2 - CaO
SYSTEM WHOSE MELTING
POINTS ARE 1500°C
OR LOWER

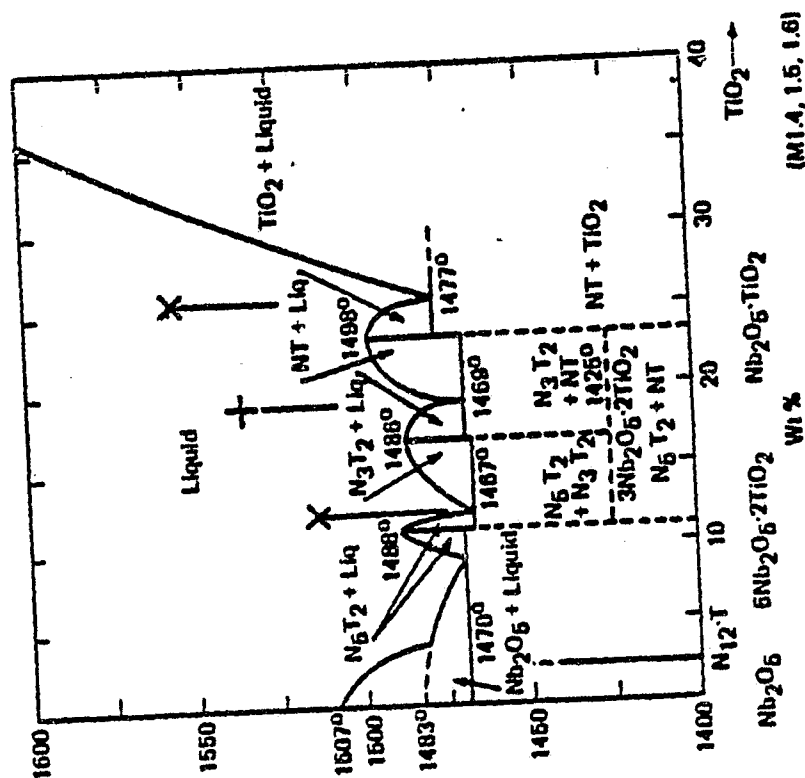


$\text{Nb}_2\text{O}_5\text{--CaO}$



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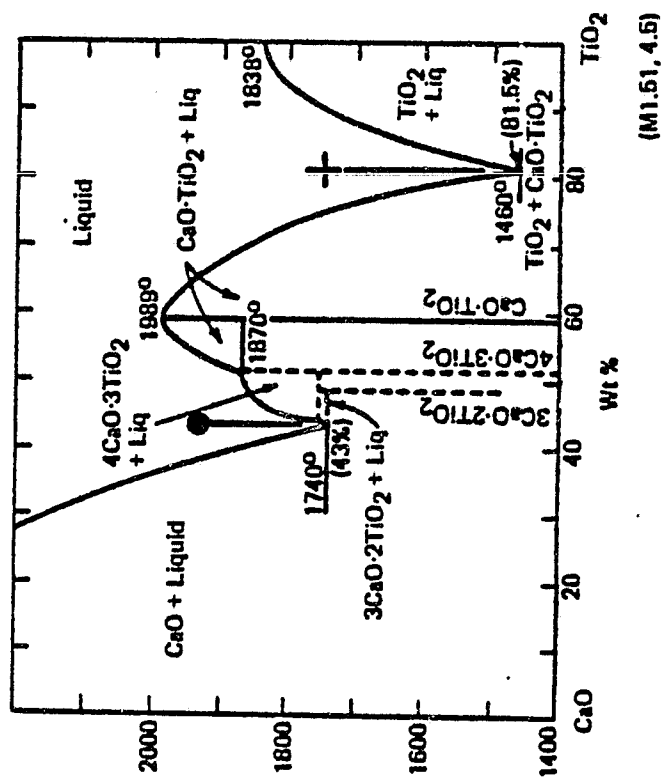
$\text{TiO}_2\text{--Nb}_2\text{O}_5$



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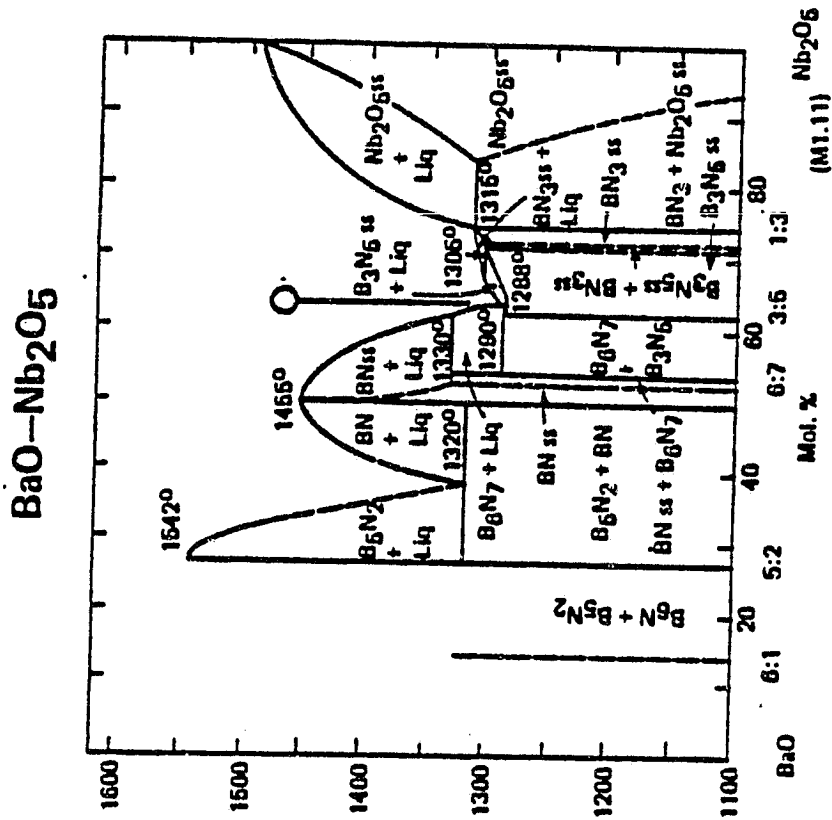
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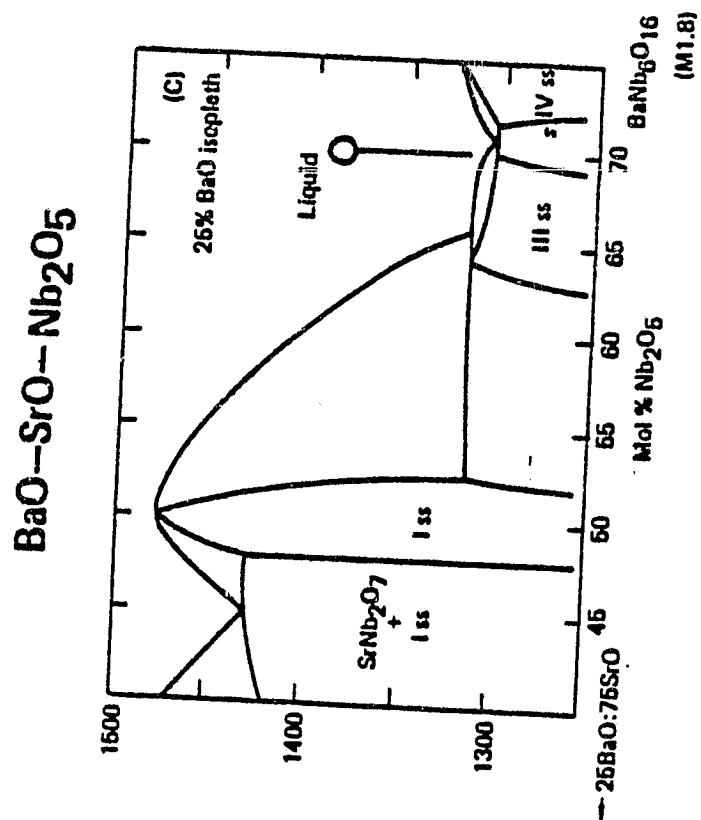
$\text{CaO}-\text{TiO}_2$



(M1.51, 4.5)

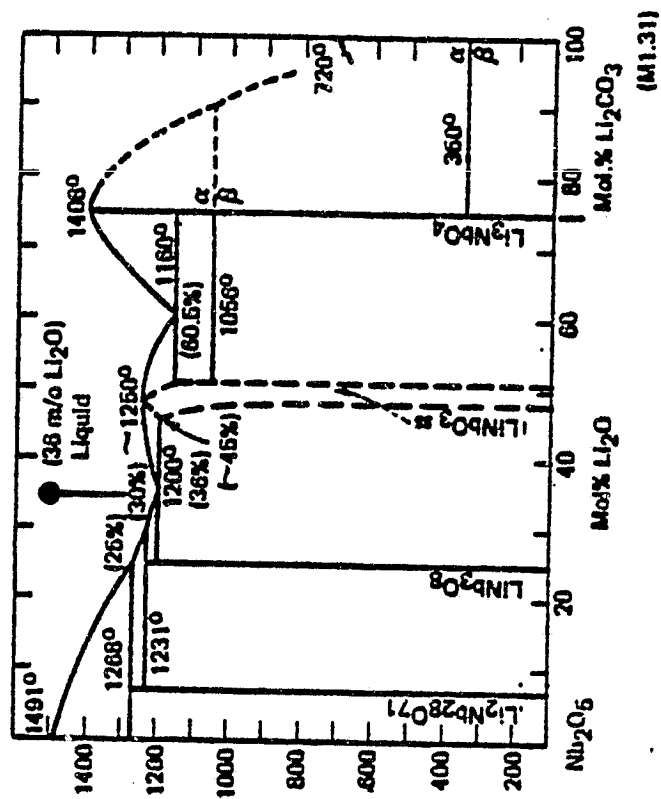
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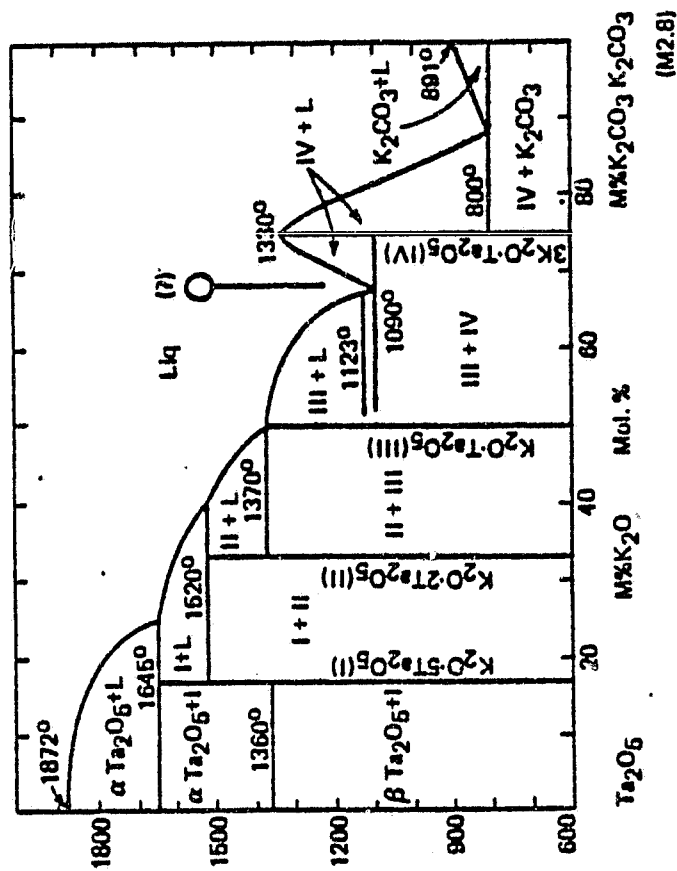
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$\text{Nb}_2\text{O}_5\text{--Li}_2\text{CO}_3$



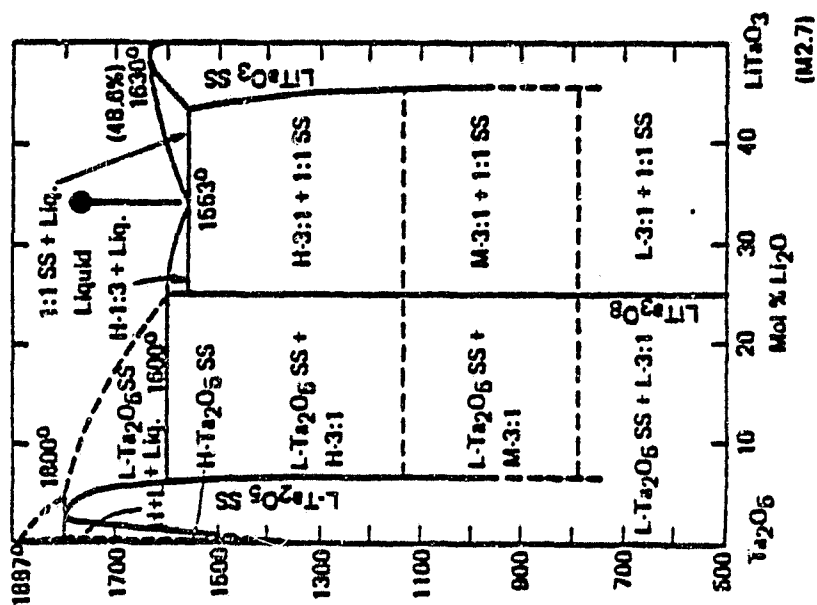
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$\text{Ta}_2\text{O}_5\text{--K}_2\text{O (OR K}_2\text{CO}_3\text{)}$



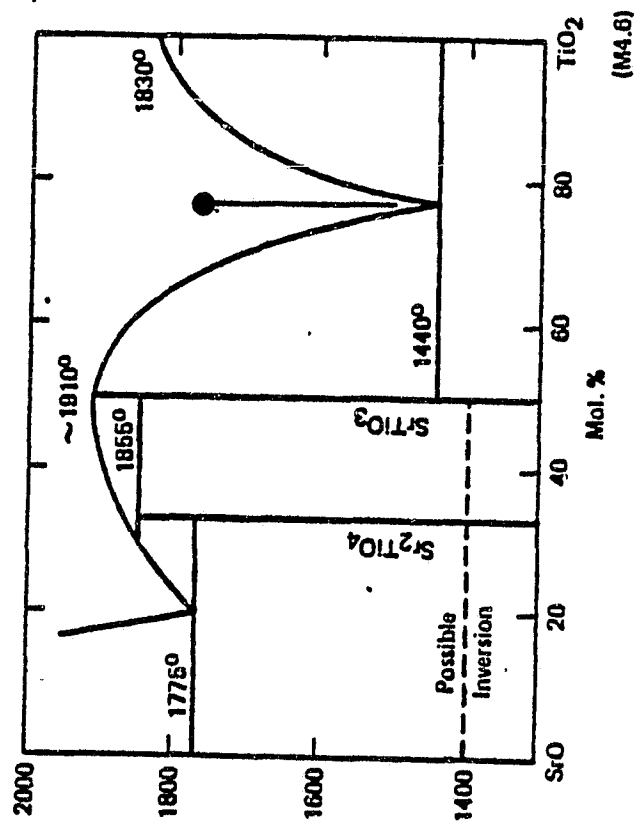
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$\text{Ta}_2\text{O}_5\text{--Li}_2\text{O}$



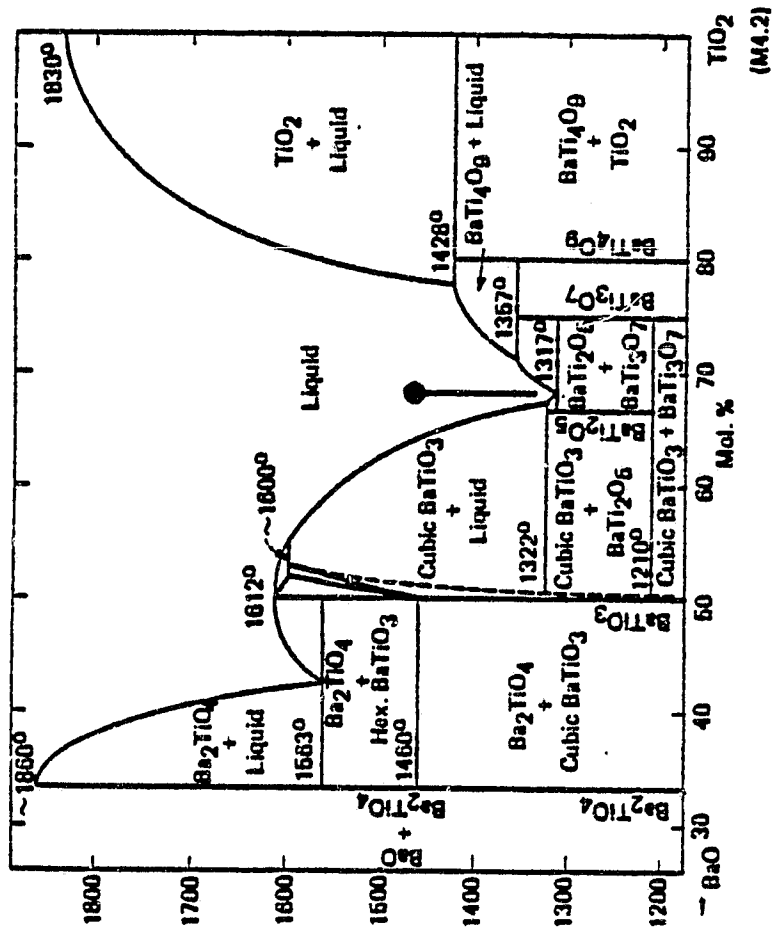
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SrO-TiO₂

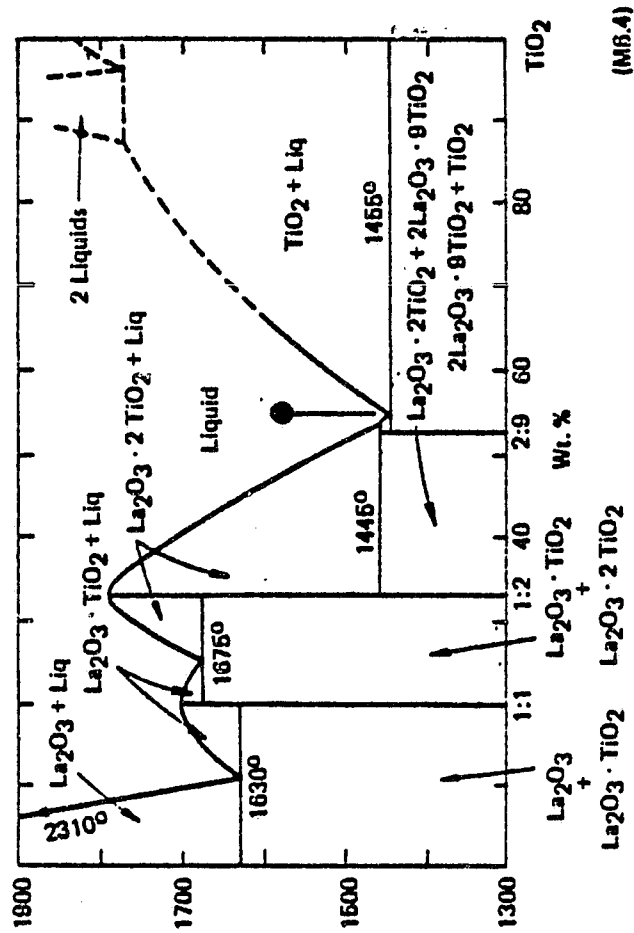


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BaO-TiO₂



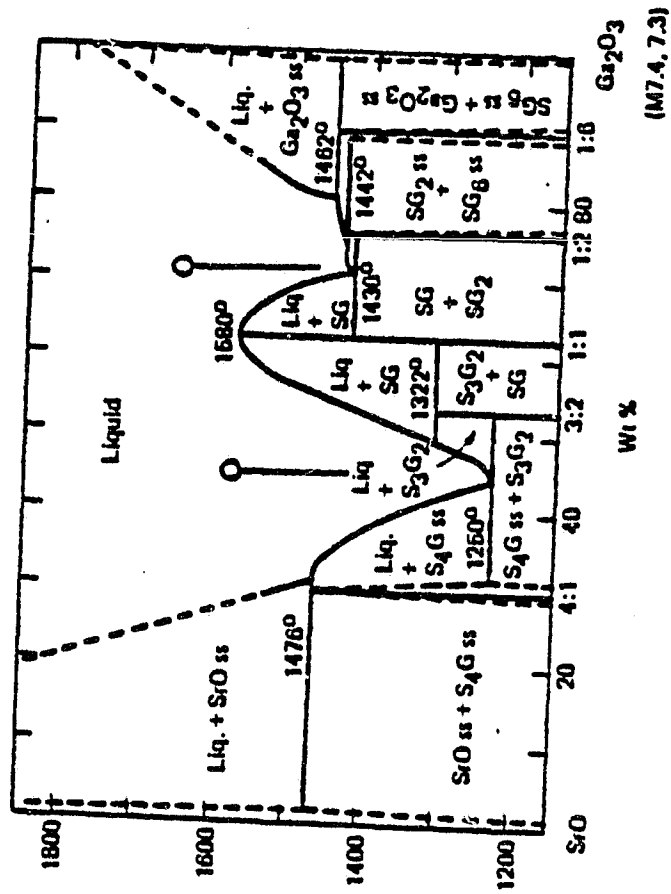
SYSTEM $\text{La}_2\text{O}_3\text{--TiO}_2$



(M6.4)

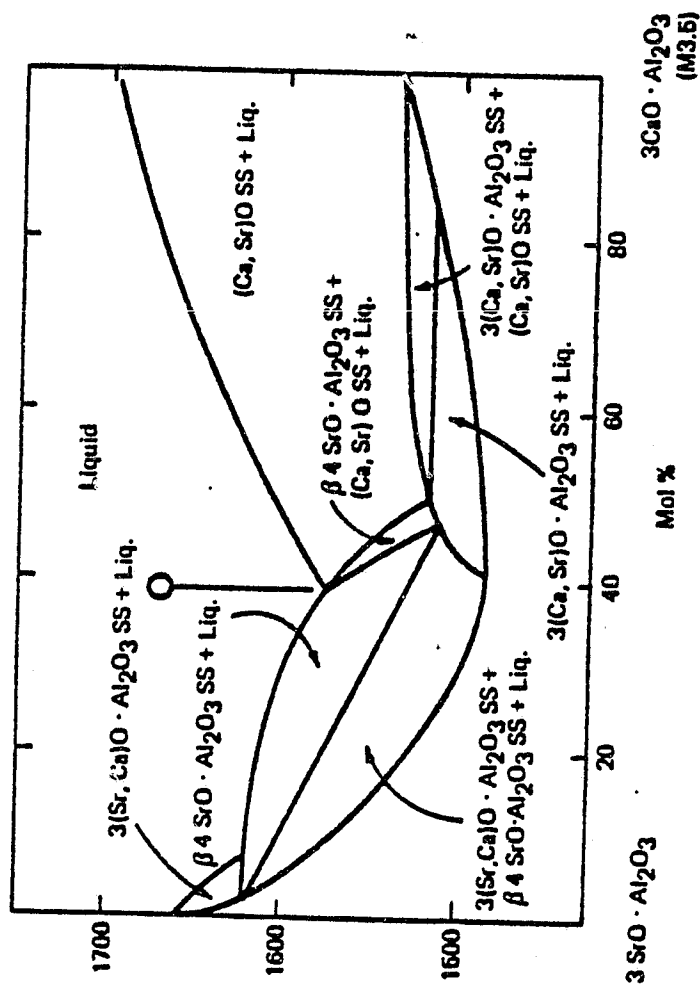
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SrO-Ga₂O₃



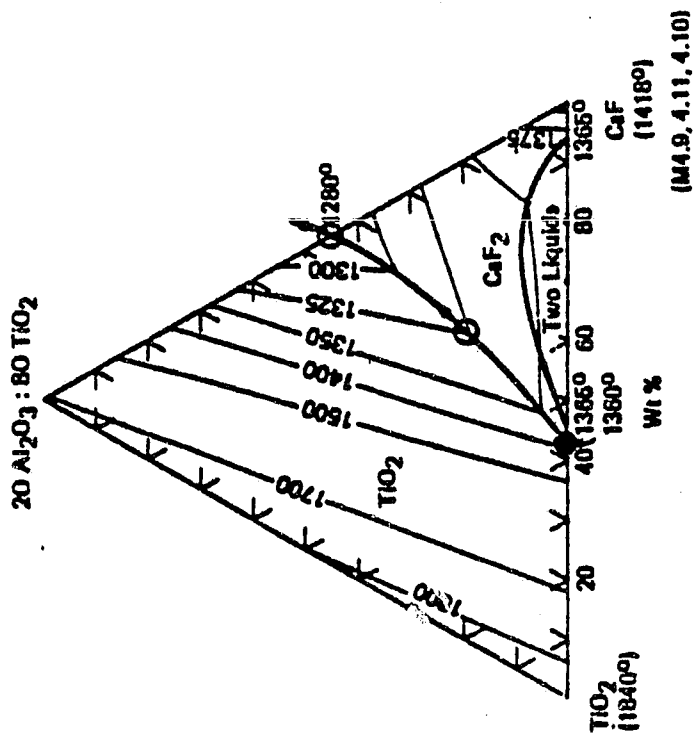
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PSEUDOBINARY SYSTEM $3\text{CaO} \cdot \text{Al}_2\text{O}_3 - 3\text{SrO} \cdot \text{Al}_2\text{O}_3$



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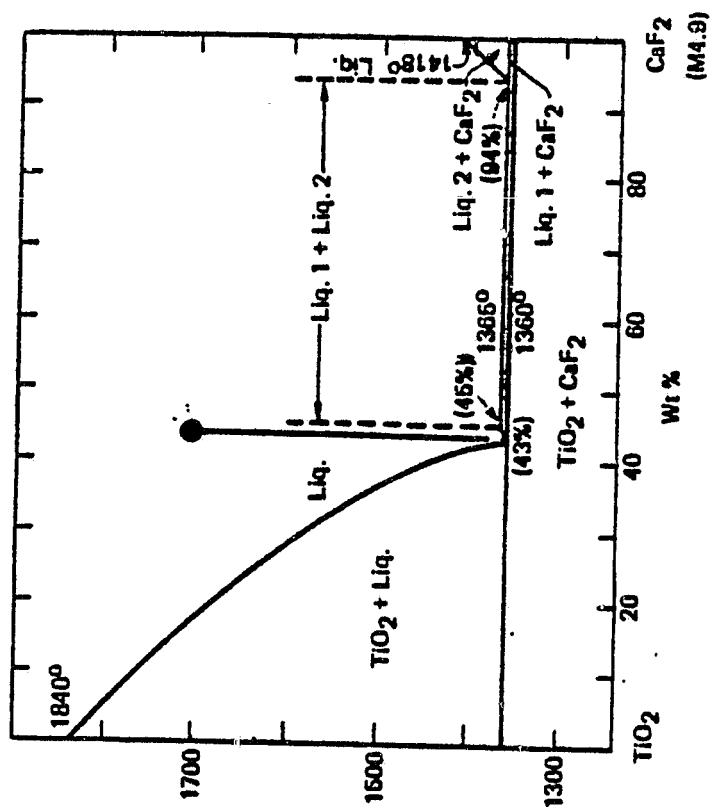
$\text{TiO}_2\text{--Al}_2\text{O}_3\text{--CaF}_2$



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$\text{TiO}_2\text{--CaF}_2$



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OUTLINE - SCIENCE REVIEW 3/19/80

- I OBJECTIVES
- II RATIONALE FOR SPACE PROCESSING OF OPTICAL GLASSES
- III INVESTIGATION PLAN
- IV SCREENING RESULTS
- V NEXT YEAR'S WORK

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NEXT YEAR'S WORK

MEA-1 (OFT)

- PREPARE MEA-1 FLIGHT SAMPLES (GALLIA-CALCIA)

SHUTTLE/PALLET EXPERIMENTS - GROUND RESEARCH

- CaF_2 ADDITIONS
- DEFINE CONTAINERLESS GLASS FORMING REGIONS
- OPTICAL PROPERTIES
- BEGIN FLIGHT SAMPLE PREPARATION TECHNIQUE DEVELOPMENT

MEA - 1

EXPERIMENT READINESS REVIEW

EXPERIMENT 77F095

CONTAINERLESS PREPARATION

OF

ADVANCED OPTICAL GLASSES

R. A. HAPPE, PRINCIPAL INVESTIGATOR

ROCKWELL INTERNATIONAL

JUNE 4, 1980

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APPENDIX A



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OBJECTIVES

EXPERIMENT 74-42/77F095

OVERALL

- O DEMONSTRATE THE FEASIBILITY OF PREPARING UNIQUE*GLASS BY CONTAINERLESS MELTING IN A NEAR-ZERO GRAVITY ENVIRONMENT.
- O DEVELOP FLIGHT HARDWARE TO ACCOMPLISH THE ABOVE.

- O SOUNDING ROCKET
- O SPACE SHUTTLE

SPAR VI

- O EVALUATE PERFORMANCE OF FLIGHT HARDWARE DURING ACTUAL FLIGHT CONDITIONS.
 - O SILICA-MODIFIED GALLIA-CALCIA.

SPAR IX

- O DEMONSTRATE THAT HARDWARE DEFICIENCIES REVEALED BY THE SPAR VI FLIGHT EXPERIMENT HAVE BEEN ELIMINATED.
- O EVALUATE PERFORMANCE IN AREA(S) WHERE NO EVALUATION WAS POSSIBLE DURING THE SPAR VI EXPERIMENT--I.E., CONTAINERLESS COOLING OF THE SPECIMEN DURING AND AFTER INSERTION OF THE COOLING SHROUD.
- O DEMONSTRATE THAT THE HARDWARE IS READY TO ACCOMPLISH THE OVERALL OBJECTIVE.

MEA-1

- O DEMONSTRATE THE FEASIBILITY OF PREPARING THE BINARY GALLIA-CALCIA GLASS BY CONTAINERLESS MELTING IN A NEAR-ZERO GRAVITY ENVIRONMENT.
- O BEGIN THE DETERMINATION OF THE CONDITIONS NECESSARY TO PREPARE A HOMOGENOUS/OPTICAL QUALITY GLASS OF LOW VISCOSITY DURING SPACE PROCESSING.

*A GLASS WHOSE VISCOSITY IS TOO LOW TO PERMIT PREPARATION TO AN OPTICAL QUALITY LEVEL BY ANY KNOWN TERRESTRIAL TECHNIQUE.

APPENDIX A

74-42 EXPERIMENT HARDWARE REQUIREMENTS

- 0 HEAT SAMPLE WELL ABOVE MELTING POINT ($>>1320^{\circ}\text{C}$)
- 0 HOLD AT TEMPERATURE LONG ENOUGH TO ACHIEVE COMPLETE MELTING
- 0 COOL RAPIDLY TO BELOW T_g ($\sim 800^{\circ}\text{C}$)
- 0 NO CONTACT DURING ENTIRE MELTING & COOLING CYCLE
- 0 FLIGHT EXPERIMENT MOTION PICTURES
- 0 NO SURFACE CONTAMINATION WITH INSOLUBLE CRYSTALLINE MATERIAL

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APPENDIX A

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STARTING SAMPLE PREPARATION METHODS

0 CASTING

0 LASER/AIR SUSPENSION MELTING

0 FLUID QUENCHED DROPLETS

CASTINGS - POTENTIAL PROBLEMS

0 SHRINKAGE POROSITY

0 ENTRAPMENT OF DIAMOND DUST

0 EXPERIMENTAL APPROACH

0 LASER REMELT DIAMOND GROUND SAMPLE

0 IF ABOVE SHOWS THERE IS A PROBLEM

0 HEAT TO 1000 TO 1275 C IN OXYGEN - HOLD
SEVERAL HOURS

0 LASER MELT

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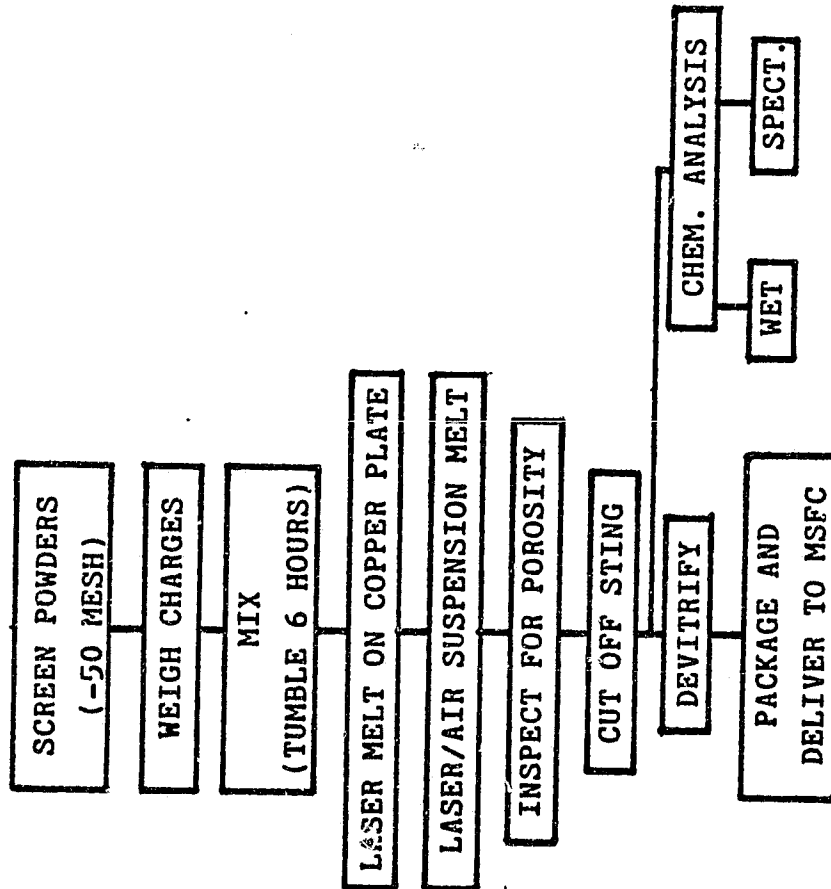
APPENDIX A

APPENDIX A

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PREPARATION PROCEDURE - LASER MELTED CHARGES



MEA-1 POST-FLIGHT ANALYSIS

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- ☐ VISUAL INSPECTION FOR GLASS FORMATION
- ☐ CHECK STRIAE (GROUND AND POLISHED PARALLEL FLATS)
 - ☐ COMPARE WITH LASER MELTED SAMPLES
 - ☐ COMPARE WITH SPAR VI AND IX SAMPLES
- ☐ MICROPROBE ANALYSIS
- ☐ OTHER

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PROBLEMS-OPEN ISSUES

0 CO₂ LASER AVAILABILITY

0 RELIABILITY OF TUNNEL LASER

0 SAFETY PROBLEMS

MEA

EXPERIMENT REVIEW

EXPERIMENT 77F095

CONTAINERLESS PREPARATION

OF

ADVANCED OPTICAL GLASSES

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R. A. HAPPE, PRINCIPAL INVESTIGATOR

ROCKWELL INTERNATIONAL

FEBRUARY 24, 1981



OBJECTIVES

EXPERIMENT 74-42/77F095

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OF POOR QUALITYOVERALL

- 0 DEMONSTRATE THE FEASIBILITY OF PREPARING UNIQUE*GLASS BY CONTAINERLESS MELTING IN A NEAR-ZERO GRAVITY ENVIRONMENT.
- 0 DEVELOP FLIGHT HARDWARE TO ACCOMPLISH THE ABOVE.

- 0 SOUNDING ROCKET
- 0 SPACE SHUTTLE

SPAR VI

- 0 EVALUATE PERFORMANCE OF FLIGHT HARDWARE DURING ACTUAL FLIGHT CONDITIONS.
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- 0 DEMONSTRATE THAT THE HARDWARE IS READY TO ACCOMPLISH THE OVERALL OBJECTIVE.

MEA-1

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- 0 BEGIN THE DETERMINATION OF THE CONDITIONS NECESSARY TO PREPARE A HOMOGENOUS/OPTICAL QUALITY GLASS OF LOW VISCOSITY DURING SPACE PROCESSING.

*A GLASS WHOSE VISCOSITY IS TOO LOW TO PERMIT PREPARATION TO AN OPTICAL QUALITY LEVEL BY ANY KNOWN TERRESTRIAL TECHNIQUE.

WHY GALLIA-CALCIA SELECTED FOR SPACE

- 0 LOW MELTING TEMPERATURE
- 0 GOOD GLASS FORMER WITH TERRESTRIAL CONTAINERLESS PREPARATION
- 0 POTENTIALLY INTERESTING OPTICAL PROPERTIES
- 0 GLASS CANNOT BE PREPARED IN USEFUL SIZES TERRESTRIALLY

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EXPERIMENT HARDWARE REQUIREMENTS

- 0 HEAT SAMPLE WELL ABOVE MELTING POINT ($>>1320^{\circ}\text{C}$)
- 0 HOLD AT TEMPERATURE LONG ENOUGH TO ACHIEVE COMPLETE MELTING
- 0 COOL RAPIDLY TO BELOW T_G ($\sim 800^{\circ}\text{C}$)
- 0 NO CONTACT DURING ENTIRE MELTING & COOLING CYCLE
- 0 FLIGHT EXPERIMENT MOTION PICTURES
- 0 NO SURFACE CONTAMINATION WITH INSOLUBLE CRYSTALLINE MATERIAL



OBJECTIVES --- FIRST HEA SCIENCE FLIGHT

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- CONFIRM CONTAINERLESS GLASS FORMATION OF BINARY GALLIA-CALCIA IN LOW G
- OBTAIN INFORMATION ON MIXING IN A LOW VISCOSITY GLASS SAMPLE WHEN CONTAINERLESS MELTED ONE HR. AT 1575 C IN LOW G

APPENDIX A

SPAR VI HIGHLIGHTS

- SAMPLE COMPOSITION - 68 w/o Ga_2O_3 - 18 w/o CaO - 14 w/o SiO_2 ($39\text{Ca}_2\text{O}_3$, 36CaO , 25SiO_2)
- FLIGHT MOTION PICTURES ACCEPTABLE
- SONIC POSITIONING SYSTEM PARTLY SUCCESSFUL
 - CONTAINERLESS POSITIONING FOR 27 SECONDS
 - SAMPLE ATTACHED TO CAGE WIRE --- REMAINED ATTACHED
BALANCE OF CYCLE
- TEMPERATURE/TIME CYCLE SATISFACTORY
 - NO EVIDENCE OF INCOMPLETE MELTING IN RETURNED SAMPLE
- SAMPLE SPHERICAL (EXCEPT AT CONTACT AREAS WITH Pt WIRE)
- LOW RESIDUAL STRESS IN SAMPLE
- SURFACE CRYSTALLIZATION ON COOLING

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SPAR VIII HIGHLIGHTS

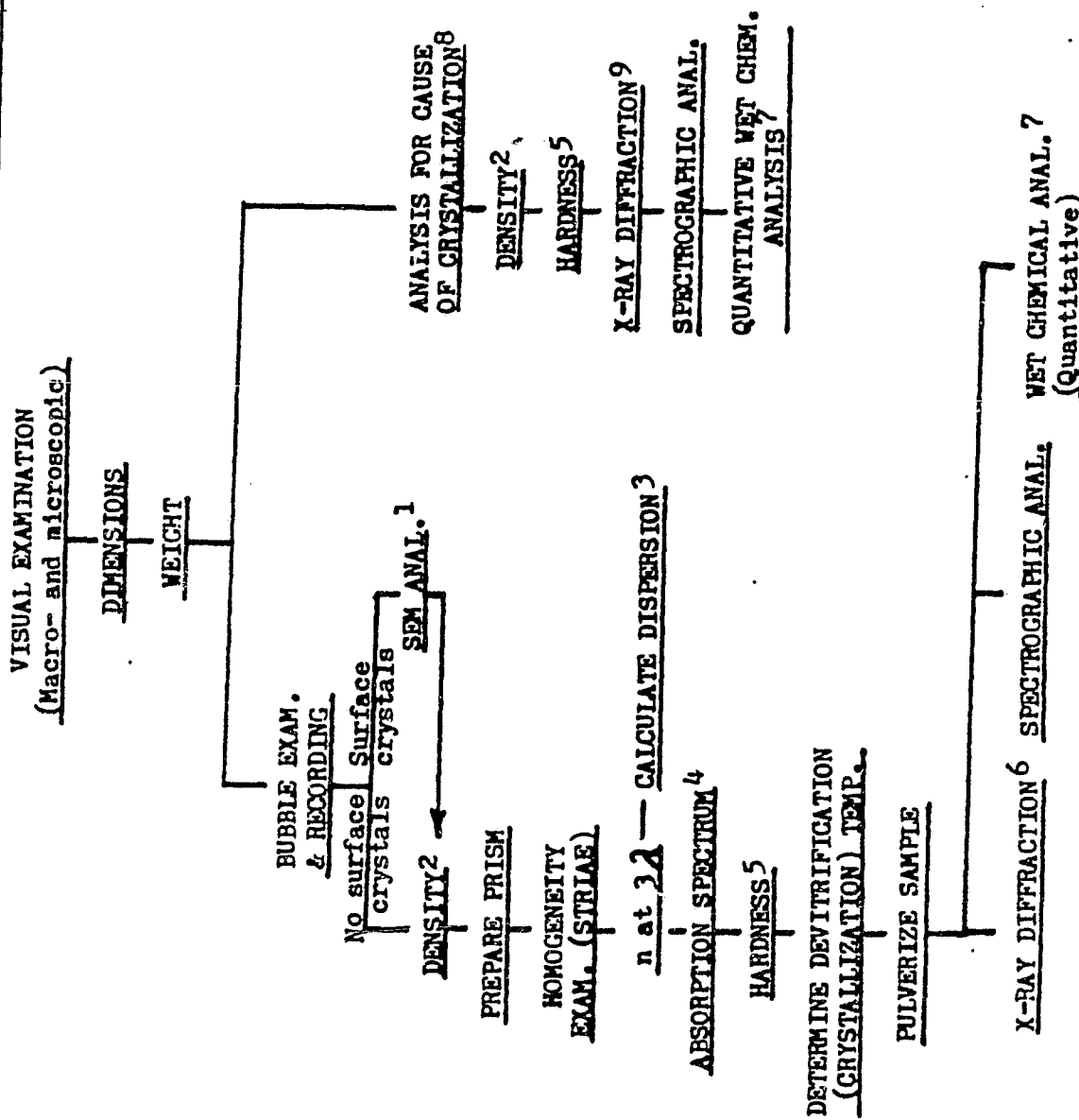
- SAMPLE COMPOSITION - 68 w/o Ga_2O_3 - 18 w/o CaO - 14 v/o SiO_2 ($39\text{Ga}_2\text{O}_3$, 36CaO , 25SiO_2)
- EXPERIMENT HARDWARE DESTROYED BY IMPACT
- SAMPLE NOT RECOVERED AS OF 2/18/81
- FILM RECOVERED FROM IMPACT HOLE AND DEVELOPED
 - SONIC POSITIONING SYSTEM PARTLY SUCCESSFUL
 - CONTAINERLESS POSITIONING FOR 90 SECONDS
 - SAMPLE ATTACHED TO CAGE WIRE --- REMAINED ATTACHED
BALANCE OF CYCLE
- Pt CONTAMINATION OF SPECIMEN --- NO CONCLUSION
- COMPLETE MELTING OF SPECIMEN --- NO CONCLUSION*
- INSERTION OF COOLING SHROUD --- NO CONCLUSION

* Probably O.K. based on telemetry data

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POST-FLIGHT ANALYSIS PLAN - FIRST MSG SCIENCE FLIGHT

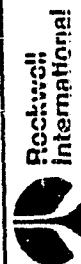


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FOOTNOTES - POST-FLIGHT ANALYSIS

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- (1) Qualitative composition of the matrix, crystals, and nucleator
- (2) Direct measurement
- (3) In blue, yellow, and red portions of the spectrum - dispersion calculated according to the general formula $V_y = \frac{n_y - 1}{n_b - n_r}$ where:
 $y =$ yellow
 $b =$ blue
 $r =$ red
- (4) Transmission will be measured over the range, $0.33\mu(3300 \text{ \AA})$ to $0.8\mu(8000 \text{ \AA})$
- (5) Mohl's scale
- (6) To confirm that sample is indeed glassy (amorphous)
- (7) For principal constituents, i.e. Ca_2O_3 and CaO
- (8) Tools - SEM, optical exam, etc.
- (9) To identify crystal types and number of major phases present.

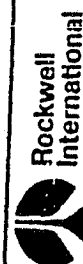


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PROBLEMS/OPEN ISSUES

- SPAR VIII FLIGHT SAMPLE
- FLIGHT HARDWARE PERFORMANCE
- SILICA PICK-UP DURING LASER/STING MELTING



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THE SILICA PICK-UP PROBLEM

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- SILICA AND GALLIA LOST BY VAPORIZATION DURING LASER MELTING
- SILICA PICKED UP FROM STING BY SOLUTION
 - NET INCREASE IN SILICA CONTENT
 - NET DECREASE IN GALLIA CONTENT
- 100% GLASS PREPARED IN 6mm SIZE IN PAST WITH 1.4 to 2.5% SILICA
- 100% GLASS PREPARED IN 1mm SIZE WITH 0% SILICA
- CURRENT STING MELTING PRACTICE GIVES 4 TO 7 % SILICA
- SAMPLES WITH 2.7 % SILICA LASER MELTED IN CONTACT WITH COLD AL OR CU WERE CRYSTALLINE
- 4 % SILICA GLASS SAMPLES PREVIOUSLY STING MELTED REMAINED GLASSY DURING REHEATING ON COLD AL OR CU

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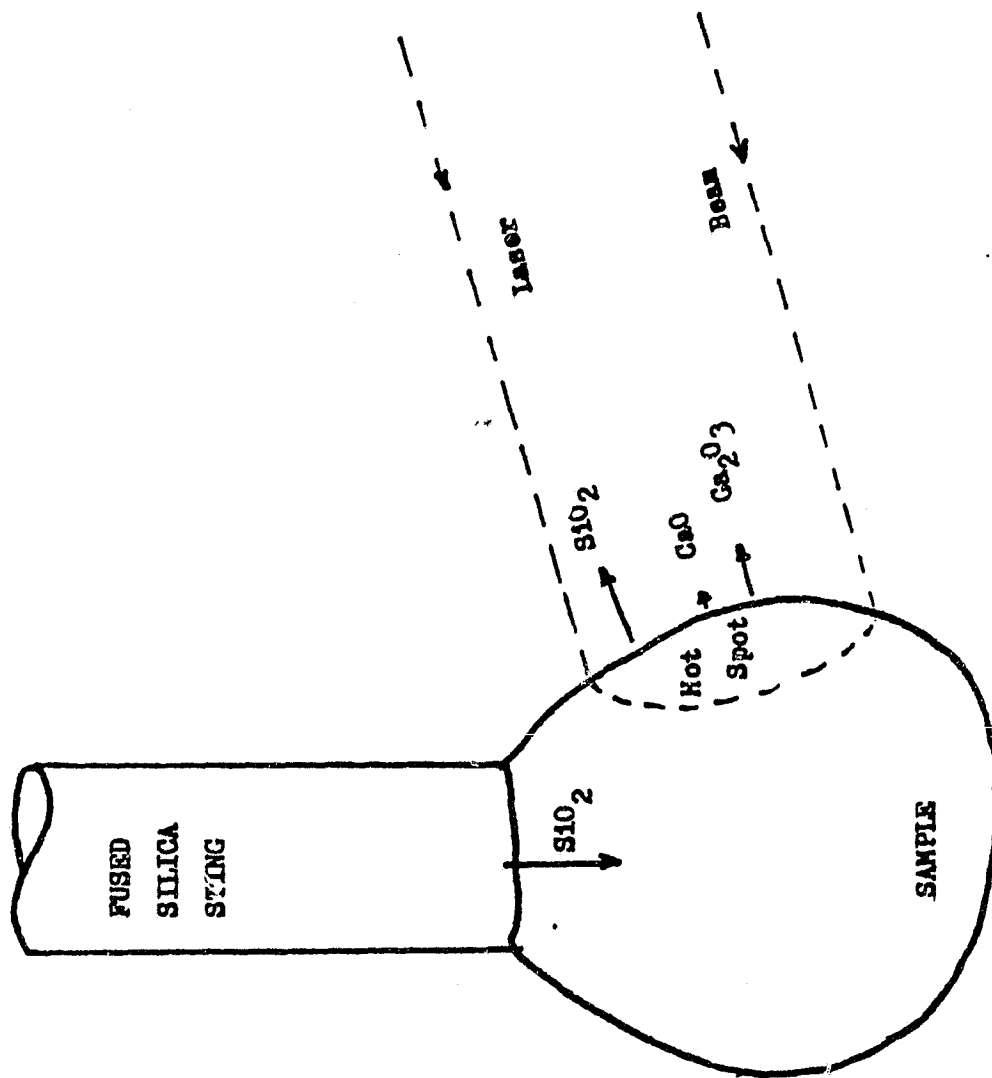


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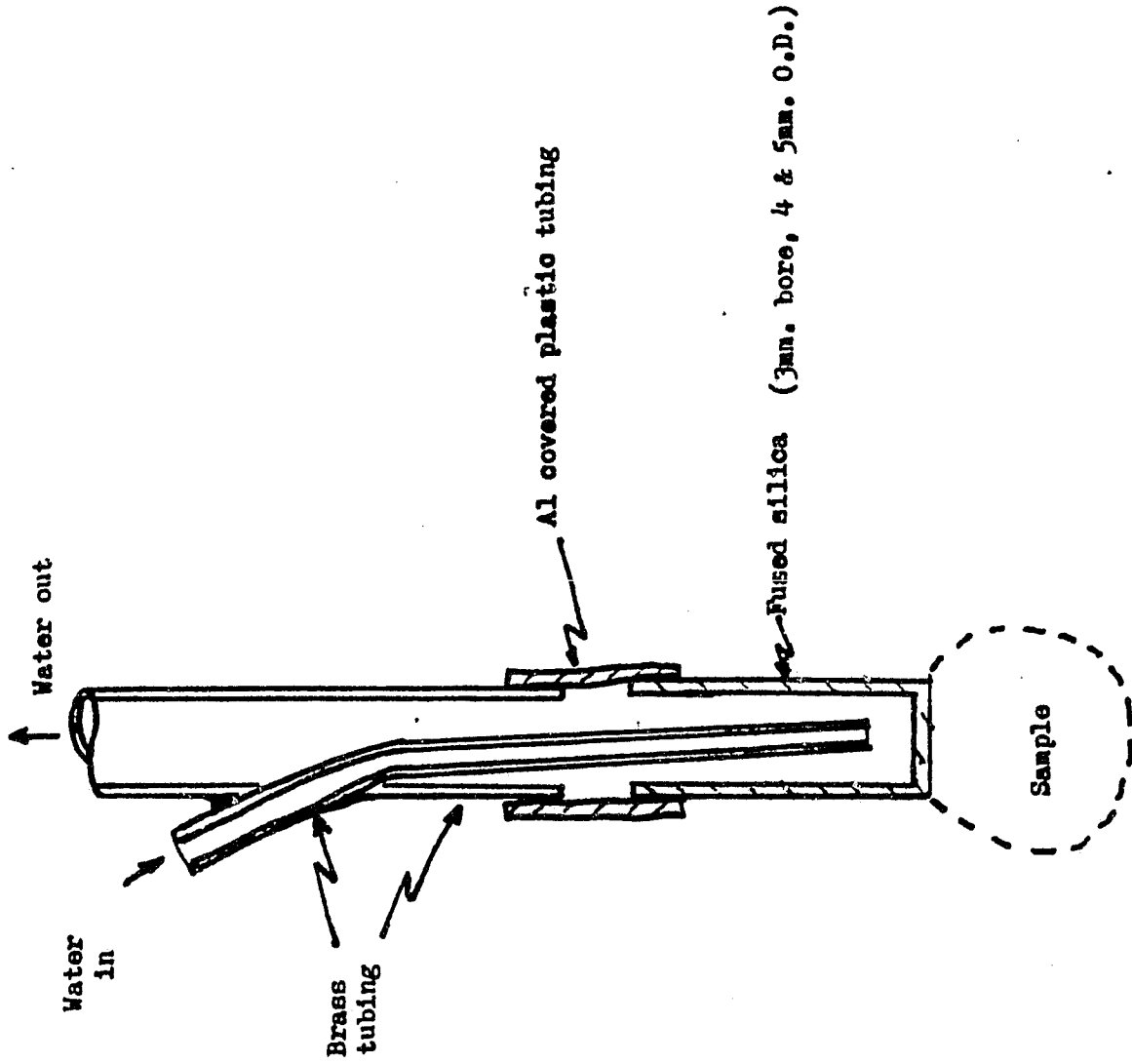
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WATER-COOLED STING



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FUTURE WORK

- COMPLETE WORK ON SILICA PICK-UP
- EXPERIMENTS TO REDUCE SILICA PICK-UP
 - WATER-COOLED STING
 - NEW CO₂ LASER AT SCIENCE CENTER
 - SUSPENSION WITHOUT SILICA STING - ??
- PREPARE BETTER SAMPLES FOR FIRST MEA SCIENCE FLIGHT
- RESUME WORK ON NEW COMPOSITIONS
 - RE-MAKE, WITH LOW SILICA PICK-UP, SAMPLES THAT WERE GLASS-FORMERS DURING INITIAL SCREENING
 - COMPLETE SCREENING

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REV A

EXPERIMENT REQUIREMENTS AND
IMPLEMENTATION PLAN (ERIP)
FOR EXPERIMENT NO. 042
GLASS FORMATION --
MEA HIGH-TEMPERATURE SINGLE-AXIS
ACOUSTIC LEVITATOR (MISSION MEA-1)

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
GEORGE C. MARSHALL SPACE FLIGHT CENTER
MARSHALL SPACE FLIGHT CENTER, ALABAMA

REVISED JULY 1981

PREPARED BY K.S. KIM

PRINCIPAL INVESTIGATOR: R.A. HAPPE
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CONTRACT NAS8-32953



Rockwell International
Space Operations and
Satellite Systems Division



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1. INTRODUCTION

This document describes, from the Principal Investigator's (PI's) point of view, the implementation of one MEA flight experiment. If the flight experiment is completely successful, three 0.6cm ($\frac{1}{4}$ inch) diameter boules of new glass compositions with potential optical applications will have been prepared and a solid sphere levitated for 5 minutes. The glass samples consist of two ternary mixtures (39.3 Ga_2O_3 :35.7 CaO : 25 SiO_2 (mol %)) and a eutectic binary composition (56 Ga_2O_3 :44 CaO (mol %)). An alumina sphere has been chosen as the solid sample for purely levitational purpose. How this glass experiment fits into the overall space glass program is described in the next section.

2. BACKGROUND

The combination of circumstances attending the melting of materials in space presents the possibility of making glasses from substances that, to date, have been observed only in the crystalline condition. A solid container is not needed during the melting and superheating portion of the manufacturing cycle. The only contact of the melt with its surroundings can therefore be a gaseous atmosphere or, if so desired, a vacuum. Thus, it is possible to melt many high-melting-point materials that heretofore could not be successfully melted because of reaction with the crucible material. This advantage alone might permit the preparation of new substances as glasses if their viscosity becomes sufficiently high on cooling to suppress crystal growth.

In addition, space melting permits cooling without the use of a solid mold. Thus many of the usual crystal nucleation sites are eliminated. Unless a given material can spontaneously nucleate on cooling, undercooling below the normal melting point will occur. If a sufficient amount of undercooling is accompanied by a sufficient increase in viscosity, crystallization will be avoided entirely and glass will result.

Studies of space glasses by the PI to date have been confined to oxide glasses. It is felt that many of the principles that will evolve from a study of oxide glasses, which can be melted in air, permit a more direct approach to nonoxide materials in the future.

During the course of terrestrial research conducted by the PI over the past several years (NASA contracts NAS8-28014 and NAS8-28991), it has been shown that new glasses with potentially interesting optical properties can be

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prepared using a containerless melting technique. The technique most recently used for terrestrial research consists of suspending a molten drop of oxide material on a laminar flow air stream. The energy for melting is supplied by a 1½ kW output CO₂ laser. The molten sample is steadied in the air stream by contacting it with a fused silica (glass) probe, or sting. The details of the techniques used and the results obtained are given in References 1 and 2.

Using the techniques described, several new optical glasses have been prepared in the 0.6cm (¼ inch) diameter size. Among them is the gallia-calcia glass, which is one of the flight specimens described in this document.

2.1 RATIONALE BEHIND THE OVERALL APPROACH TO GLASS MAKING IN SPACE

The concept of glass making in space is of interest for the following reasons:

1. There is a strong possibility that oxides such as La₂O₃, Ta₂O₅, Nb₂O₅, Al₂O₃, Ga₂O₃, and Y₂O₃, and some of the rare earth oxides can be prepared as glasses in spherical boules of commercially useful sizes through space melting and cooling.
2. Glasses produced from such oxides or combinations of them with other oxide additions should have optical properties not obtainable in the conventional silicate-, borate-, and phosphate-based glasses.
3. The combination of optical properties projected for such glasses should make them suitable for use in advanced optical systems.
4. The spherical shape of glass boules that would result naturally from space production is quite suitable for the making of lenses and windows.

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5. The production of glass, given a well-engineered space facility, should be well within the capabilities of the astronauts after a suitable brief training period, the more technically and skill-oriented operations being performed terrestrially before and after flight operations.
6. A well-directed research and development program leading to the production of useful space glasses should yield much valuable scientific information on the nature of glass formation, nucleation theory, etc.

It is envisioned that the process described need not be limited to oxide glasses. Perhaps other compounds, such as carbides, nitrides, and chalcogenides, will ultimately be producible as glasses by space processing.

2.2 APPLICATIONS - OPTICAL GLASSES

An Abbe diagram, familiar to lens designers, is shown in Figure 1. The index of refraction in sodium D light is plotted on the ordinate and the Abbe number, an inverse measure of dispersion, is plotted on the abscissa. The higher Abbe numbers to the left of the diagram indicate a low dispersion (i.e., a flatter slope of the index versus wavelength curve), and the lower Abbe numbers to the right have high dispersion, or a steeper index versus wavelength curve. A century ago, the flint glasses were developed. This permitted the designing of the first achromatic, or color-corrected, multi-element lenses. Responding to the demands for better quality lenses, the optical glass industry developed more glasses with properties between those of the crown and flint glasses. More recently, glasses have been developed to fill

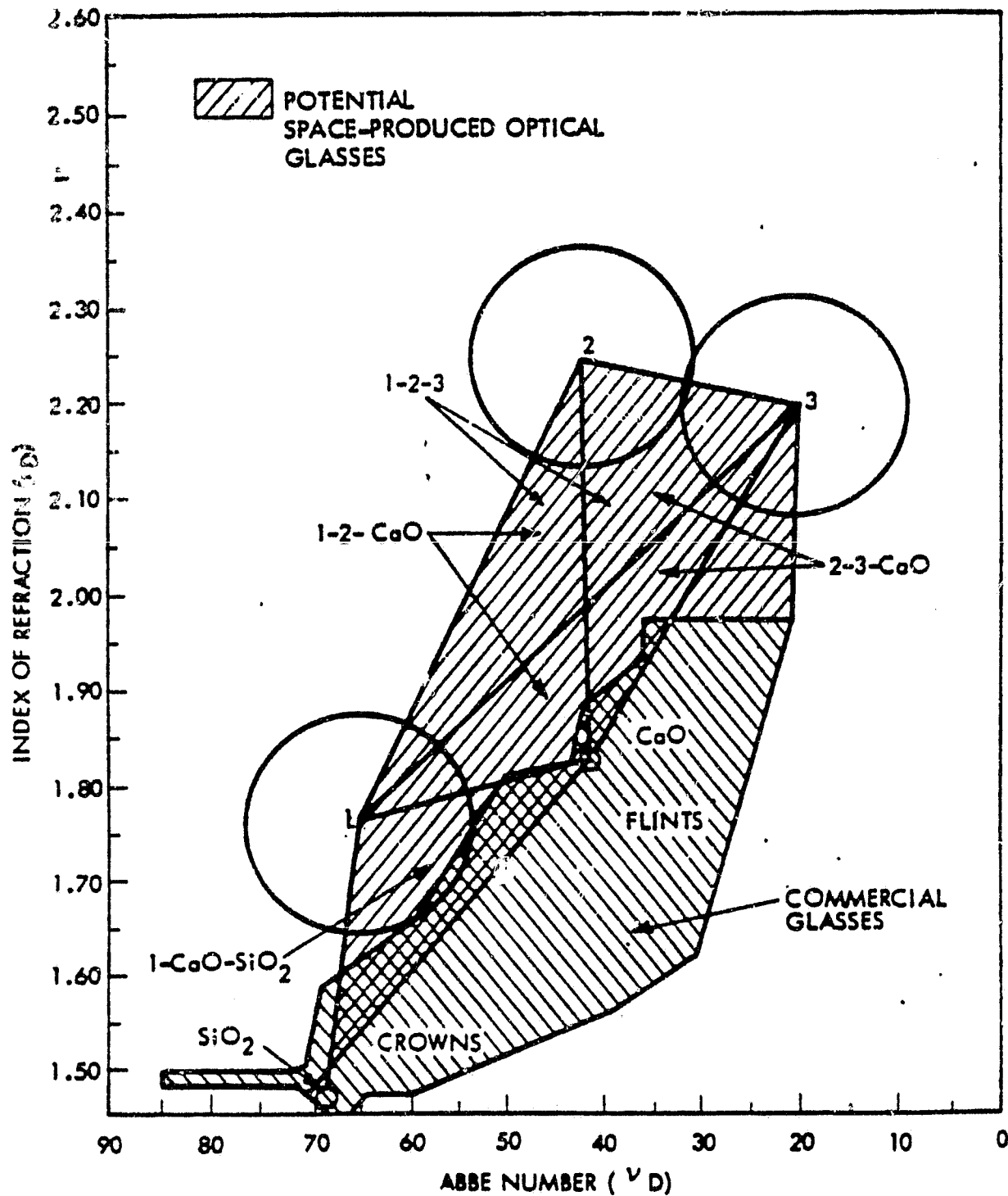


Figure 1. Potential Space-Produced Optical Glasses

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out the area labeled "commercial glasses" in Figure 1. The trend has been to push the area up and to the left, and many of the glasses have very complex compositions. It also should be noted that because lens design is an iterative process, isolated glasses with unusual combinations of properties are rarely of value. The lens design process requires that small shifts in optical properties from those initially selected be available.

If glasses could be prepared from oxides beyond the reach of current technology, the area of useful properties could be more than doubled, as is shown in the area labeled "potential space-produced glasses" in Figure 1. If the promise of space production can be realized by the preparation of simple compositions with properties falling roughly in the circles marked 1, 2, and 3, it should be possible, by combining the compositions, to fill in the areas shown in the figure. Thus, efforts to date have been largely devoted to attempts to prepare simple compositions with glass properties falling roughly within the three circles in Figure 1.

Additionally, there is another important area of application that new, space-produced, optical glasses might fill. Space production offers an opportunity to prepare new families of glasses free of the usual base oxides, SiO_2 , B_2O_3 , and P_2O_5 . Noncross-bred glasses should open possibilities for the lens designer for correcting "secondary spectrum." Reference 3 covers this subject in more detail.

It is expected that the compositions selected for the experiment described in this document fall into the area designated "1" in Figure 1. While they have optical properties of potential interest they were selected for a number

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of important and practical reasons as detailed in Section 3. It must be emphasized that the area of applications is large and the compositions to be shown are representative of that large area.*

Table 1, which has been abstracted from an applications study performed by the Perkin-Elmer Corporation (Reference 4), lists some of the specific types of systems in which space glasses might find application. The reader is referred to Appendix I of Reference 4 for a comprehensive treatment of applications for space optical glasses.

Table 1. Potential Applications for
Space-Produced Glasses

Non-Imaging
Host materials for 1.06 micron lasers
Raw materials for coatings
Imaging
Microscope objectives
Low light level lenses
Long focal length lenses
Anastigmatic photo objectives
Aplanats
Lower curvature lenses
Zoom spectrometers
Monochrometers
Polarizing microscopes
High speed, large lenses
Oil immersion microscope objectives
Fiber optics bundles

*For a listing of oxides that are candidates for the other two areas of Figure 1, the reader is referred to Appendix I of Reference 1

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As an example of the type of application possible with space-produced glass, a telephoto lens for a 35-mm still camera can be considered. By substituting a space glass for conventional glasses in one or more elements of, let us say, a seven-element telephoto lens, it may be possible to maintain the same image quality and increase the aperture from, say, $f/2.8$ to $f/1.4$. Such an increase would expand the range of application for such a lens significantly. Under most circumstances in which telephoto lenses are used, the photographer has no control of lighting, the distant subjects being beyond the range of flash, or other, illumination from the camera position. The availability of such a lens would make it possible, for example, for the photographer to increase the shutter speed by a factor of four, a significant advantage in using a hand-held telephoto lens or where the subject is in motion.



3. EXPERIMENT DEFINITION

3.1 DESCRIPTION OF THE FLIGHT EXPERIMENT

The primary objectives of the first MEA flight experiment are to study the performance of the experiment hardware, especially the single-axis acoustic levitator and the automated multi-sample processing unit, under actual flight conditions and to obtain new glasses. Specifically, the experiment can be considered to consist of three subtasks:

1. Levitate four samples (one unmelted, three melted) in the furnace for specified periods of time in sequence,
2. Operate the automated multi-sample processing unit, and
3. Produce three new glass spheres.

Levitation of an unmelted sphere is to facilitate evaluation of the acoustic levitator performance. Two of the three glass spheres to be produced are of a gallia-calcia-silica composition and the third a gallia-calcia composition. Alumina has been chosen as the solid to be levitated because of its melting point being higher than the highest temperature the furnace can attain. The number of samples, the compositions, and the duration of soaking at high temperatures have been determined on the basis of the objectives stated above with particular constraints being imposed on the first MEA experiment that the total experiment time should not exceed 90 minutes, the power available is limited, and the furnace and other automated mechanisms have their characteristic response times.

3.1.1 Gallia-Calcia Experiment

The composition of approximately 56 Ga_2O_3 :44 CaO (mol %) has been chosen as one of the compositions for the glass experiment. This is the composition originally chosen for the two sounding rocket experiments but later replaced

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by the ternary gallia-calcia-silica for both experiments for reason given in the Section 3.1.2. The binary composition is near a eutectic in the phase diagram and the eutectic temperature is approximately 1323°C (See Figure 2). This composition is chosen for the following reasons:

1. It has the lowest melting temperature of any of the new optical glasses studied by the Principal Investigator to date.
2. It is a relatively good glass former in the 0.6 cm ($\frac{1}{4}$ inch, 0.8 gm) size under terrestrial, containerless, melting conditions.
3. It potentially has optical properties of interest to the optics industry.
4. Prior to the terrestrial containerless melting work mentioned above, it has not been prepared in a size exceeding 50 mg.

One sample will be of this composition and soaked at high temperatures for 9 minutes.

3.1.2 Gallia-Calcia-Silica Experiment

The composition of $39.3 \text{ Ga}_2\text{O}_3:35.7 \text{ CaO}:25.0 \text{ SiO}_2$ (mol %) has also been chosen as one of the compositions for the experiment. This is the composition that was actually used in the previous two SPAR experiments in lieu of the binary composition.

The primary reason for selecting the silica-modified gallia-calcia is to facilitate the evaluation of the equipment performance. Namely, the ternary has a much higher tendency to form a glass than the binary gallia-calcia by virtue of its higher viscosity, and so a glass would result even when it is partially melted and/or melted and cooled in contact with a container. Thus

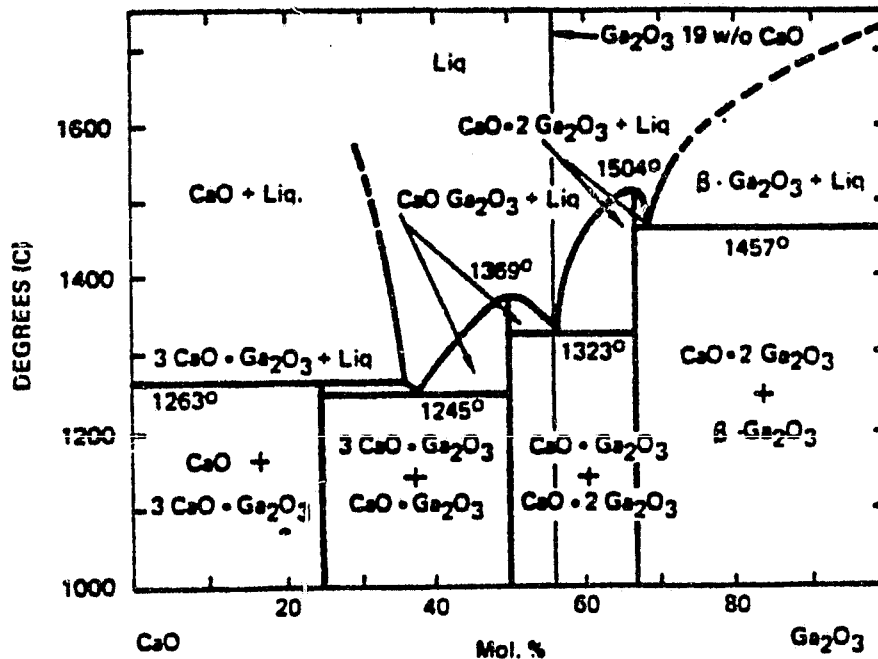


Figure 2. The CaO - Ga₂O₃ Phase Diagram (From Reference 5)

the degree of glass formation will indicate the extent to which the sample has melted. The performance of the furnace and the acoustic levitator can be evaluated from this. The 25 mol % silica content was chosen since that was found from previous experiments to be the lowest in silica among the ternary gallia-calcia-silica compositions that yielded a significant amount of glass. Two samples will be made of this composition and will be soaked at high temperatures for 9 minutes, each.

3.1.3 Alumina Experiment

Addition of this material to the group of samples to be processed in space is mainly for the purpose of evaluating the performance of the acoustic

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levitator. Alumina has a melting point of 2045°C and therefore will stay solid at the highest temperature the furnace attains (1600°C) during the flight experiment. If the acoustic levitator fails to perform properly and the specimen touches the cage, there is a much greater probability for a solid specimen to return to the sonic well and be captured there than for a molten one to do likewise. This will permit study of long term performance of the levitator. This sample will be levitated at the same high temperatures as the other specimens for 5 minutes.

3.2 OBJECTIVES

The specific objectives of the flight experiment include the following:

1. Gain experience with the performance of the flight experiment hardware; more specifically:
 - 0 Determine the effectiveness of the acoustic levitator in preventing contact of the melts with the furnace walls and holding the melts in the center of the furnace hot zone at high temperatures.
 - 0 Determine the effectiveness of the furnace, the cooling shroud, and associated hardware.
 - 0 Determine the effectiveness of the automated multi-sample insertion/retrieval and storage unit and associated hardware.
2. Gain insight into the degree of equipment complexity required to prepare glasses in zero gravity.
3. Determine whether the longer melting time available on a Shuttle flight can result in a significantly more homogeneous glass than was

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possible during the earlier rocket flights.

4. Determine the characteristics of zero-gravity-prepared glass samples.

5. Attain a position that will permit the determination of accurate optical properties of the new glasses.

3.3 PREFLIGHT (CHARACTERIZATION) TESTS

The flight starting materials (except alumina) will be characterized by the Principal Investigator in terms of containerless melting glass-forming tendencies, apparent density, chemistry, and freedom from bubble formers. A significant portion of the flight sample characterization effort will be devoted to cinematography studies of the heating and cooling of the flight sample compositions, using a laser melting/air suspension containerless melting technique with a silica probe. The films will be analyzed to determine critical temperatures and, with appropriate scaling, times for use in programming the flight experiments. The alumina sample will be characterized for density, etc., as detailed in Section 5.

3.4 FLIGHT EQUIPMENT TESTS

Suitable flight equipment tests will be specified by Interasonics and executed by Interasonics and NASA. The Principal Investigator will review the test programs and results to assure that experiment requirements are being met.

3.5 POSTFLIGHT EVALUATION

The Principal Investigator is responsible for conducting postflight evaluation of the flight results. The evaluation will consist of two principal activities:

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1. Examination of flight equipment. It is anticipated that this will be conducted jointly with Intersonics and NASA.
2. Evaluation of the flight experiment samples and comparison with preflight test results and with the earlier sounding rocket (SPAR) flight samples.

3.6 FLIGHT EQUIPMENT DESIGN AND INTEGRATION

The Principal Investigator will perform consulting services in connection with the design of the flight equipment. He will also participate in all equipment reviews by NASA and will determine the acceptability of flight hardware, ground test results, and flight programs. He will maintain cognizance of all design and flight program changes as they occur and will determine their acceptability.

4. DESCRIPTION OF EXPERIMENT SPECIMENS

4.1 COMPOSITION

4.1.1 Gallia-Calcia

In order to maximize the possibilities for obtaining a homogeneous glass, the starting material will be prepared in an essentially 100 percent eutectic structure. Such a structure is free of a primary phase and is the finest structure obtainable in this type of system. The composition of the material will be slightly different from that corresponding to the eutectic composition in the binary Ga_2O_3 -CaO phase diagram. Experiments for the SPAR program have shown that the eutectic composition shifts to a higher gallia content with rapid cooling, namely to a composition near 61 Ga_2O_3 :39 CaO (mol %). This phenomenon is known as "modification of the eutectic" (Reference 6); and the starting material will be of this composition.

4.1.2 Gallia-Calcia-Silica

Lacking a ternary phase diagram for the Ga_2O_3 -CaO- SiO_2 systems, it was assumed that a eutectic valley existed in the ternary system which extended from the 56 mol % gallia eutectic composition in the binary gallia-calcia system to the 62 mol % silica eutectic. Out of the eutectic composition range the 29.3 Ga_2O_3 :35.7 CaO 25.0 SiO_2 (mol %) composition will be used, as before, to produce loaf castings. The SPAR program showed that loaf casting was a very effective method to prepare a homogeneous structure of this material. The method of preparing the loaf castings was described in detail in the SPAR VI EXPERIMENT REPORT (Reference 7).

4.1.3 Alumina

This is a pure aluminum oxide (Al_2O_3), and a commercially available

pressed powder sample will be used.

4.2 SHAPE AND SIZE

For convenience for suspending in the acoustic levitator during the flight experiment, all flight samples will have a (roughly) spherical shape with the diameter approximately $\frac{1}{4}$ inch (0.6 cm).

4.3 RAW MATERIALS

All flight samples will be prepared, except the alumina sample, from Johnson-Matthey, Grade 1, oxide powders (99.999 + % pure) or equivalent. The alumina sample will be obtained as pressed powder.

4.4. FABRICATION

4.4.1 Gallia-Calcia

The gallia-calcia sample will be prepared using the technique of air suspension with a silica probe. Preparing a loaf casting with this material inevitably will result in crystallization of the material and creation of vacancies. These vacancies tend to impede the process of homogenization of the molten material during the flight experiment and thereby prevent formation of a glass. Specimens with an excessive amount of vacancies cannot be accepted as flight specimens. The air suspension technique provides an opportunity to prepare the material in a glass structure terrestrially. The only problem with this method, at present, is dissolution of silica from the silica sting to the material, resulting in a 2-5% silica content. A technique of cooling the sting with water to minimize the dissolution is being developed, and the flight samples to be delivered to NASA are expected to contain, if any, very little silica. The samples will be devitrified without melting

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terrestrially before they are used in the flight experiment. It should be noted that the devitrification at temperatures well below the melting point does not result in creation of an excessive amount of vacancies.

4.4.2 Gallia-Calcia-Silica

The ternary composition, silica-modified gallia-calcia sample, will be devitrified spheres cut from loaf castings, which will be prepared from pressings.

Starting pressings will be prepared by the Haselden Company of San Jose, California. High-purity graphite tooling will be used exclusively. Haselden will supply the Principal Investigator with a report describing the processing of the pressings, including mixing of the powders, chemical analysis of the tooling, pressures used, temperatures achieved, apparent density achieved, etc.. Starting castings will be prepared by the Principal Investigator. All details of the fabrication of the loaf castings and subsequent operations, which will be a close duplication of the earlier procedures for the SPAR samples (reported in Reference 7), will be recorded as a matter of course in the laboratory notebook.

4.5 QUANTITY SUPPLIED AND PACKAGING

A minimum of five samples, each, of the ternary and the binary samples, and three alumina will be supplied to NASA prior to flight.

All three types of samples are known to be quite stable in normal room atmosphere as evidenced in the SPAR program and therefore no unusual care will be required in handling the samples. Past experience has shown that the ternary and the binary compositions are impervious to moisture for over a year.

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However, there is some evidence that the performance of the acoustic levitator is significantly affected by the presence of water vapor in the levitator environment. Because of this, each flight sample container will be air-tight and contain a desiccant. Each container will bear a serial number traceable to the PI's records.

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5. PREFLIGHT (CHARACTERIZATION) TESTS OF FLIGHT SAMPLES

The following properties will be determined on representative examples of the ternary and the binary flight samples:

1. Density, dimensions, weights
2. Chemical analysis, if necessary
3. Impact resistance
4. Resistance to deterioration when exposed to a nondesiccated air atmosphere
5. Devitrification temperature
6. Glass forming tendency
7. Critical temperatures and times

The alumina sample will be examined for Test No. 1. For details of the test method/description, the Sample Handling and Analysis Plan should be referred to.

6. PERFORMANCE OF FLIGHT EXPERIMENTS

6.1 PRELAUNCH OPERATIONS

It is expected that the majority of the prelaunch operations required will be covered elsewhere by the equipment contractor, Intersonics, Inc.

The only requirements envisioned by the PI at this writing are:

1. The specimens after installation within the levitator assembly must not then be inserted into the furnace hot zone before the hot zone reaches 1250°C , for the levitator is not activated until that temp.
2. The specimens must not be exposed to shock conditions.
3. Care must be exercised to prevent contamination of the specimens by dust or oils that may be picked up in handling (no handling with bare hands).

6.2 FLIGHT OPERATIONS

The flight operations are described by the timeline chart shown in Figure 3. They consist of levitation of the alumina sphere, and melting, soaking at high temperatures, and cooling of the other three samples individually, all operations in sequence. Namely:

1. The furnace is turned on at time zero and when its hot zone reaches 1250°C the alumina sample is inserted into the hot zone, and simultaneously the acoustic levitator is activated. When it reaches 1600°C the temperature will be held constant at that temperature for 5 minutes, at which time the heater is turned off and the cooling shroud inserted. The sample remains suspended for $2\frac{1}{2}$ minutes for cooling. At the end of this period the cooling shroud is removed and the sample is retrieved by the multi-sample processing mechanism.

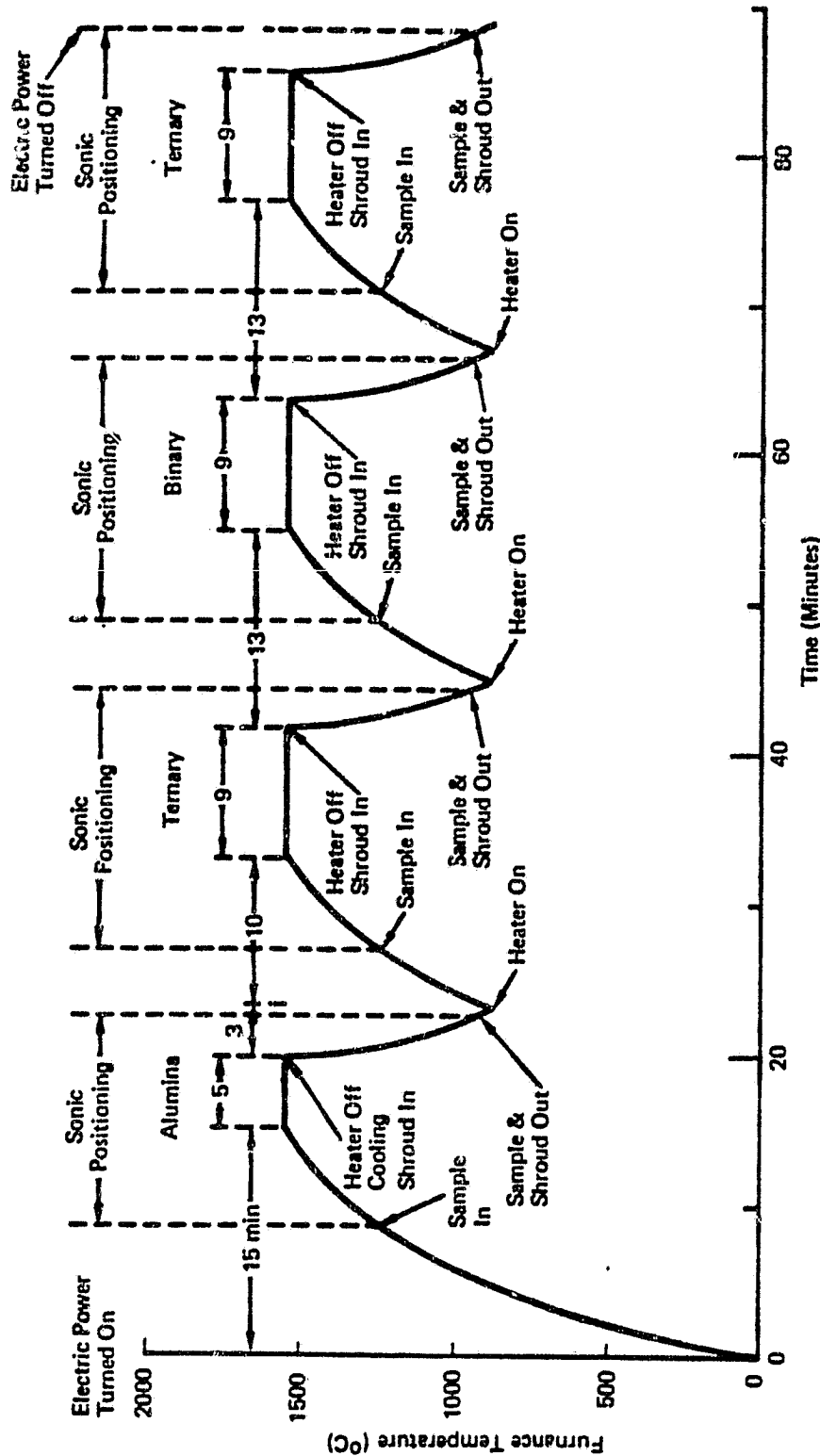


FIGURE 3. FLIGHT EXPERIMENT TIMELINE

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As soon as the sample leaves the furnace the heater is turned on.

2. The process described above will be repeated for Sample No. 2, one of the ternary samples. The entire process is identical to that for the alumina sample except that the soaking at 1600°C is 9 minutes as compared to 5 minutes for the alumina sphere.
3. The operations for Sample Nos. 3 & 4, the binary and the ternary samples, respectively, are identical to that for Sample No. 2.

In addition to the temperature cycling and the mechanical operations required, the following points should be noted:

1. The samples must not make physical contact with any crystalline material during the cycle. Examples of such prohibited crystalline material include metals and ceramics, such as furnace walls, heating elements, sample cage and dust particles. Any of these may serve as nucleating agents and prevent the attainment of a glassy state in the finished specimen. The period from the beginning of the cooling cycle to the attainment of the glass transition temperature ($\sim 800^{\circ}\text{C}$) is critical in this respect. The above requirement does not apply to the alumina sample.
2. The samples must be cooled as rapidly and uniformly as possible. Radiation cooling to an initially cooled copper shroud (less than about 250°C) should achieve the desired rapid cooling. Rapid cooling is required to maximize the possibilities for glass formation. Again, this requirement does not apply to the alumina sample.

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7. POSTFLIGHT ANALYSIS

The requirements for much of the data described in the following subsection are based on the assumption that the Principal Investigator will not be permitted to oversee the operations at the launch site.

7.1 PREFLIGHT DATA REQUIRED

The Principal Investigator shall receive, within seven days of Shuttle landing, copies of all pertinent preflight data obtained. Examples of such data include:

1. Time (date, hour, minute) specimen removed from desiccated container supplied by the PI.
2. Complete description of the specimen environment from the time removed from desiccated container to time that a temperature of 100°C (212°F) is achieved in the flight furnace. This description should include ambient temperatures ($\pm 1^{\circ}\text{C}$), relative humidities ($\pm 5\%$), dust conditions, accidental contact of the specimen with other than prescribed handling devices, etc. Motion picture coverage (100%) of the specimens from removal from the desiccated container through closing of the flight package would be highly desirable.
3. Records of furnace temperature ($\pm 30^{\circ}\text{C}$), both at the hot zone center and at the specimen location, if different, versus time from the time of furnace power turn-on to experiment shutdown. All hold periods should be indicated on the records.
4. All data on furnace atmosphere obtained, such as relative humidity or dew point.

7.2 FLIGHT DATA REQUIRED

The PI shall receive, within 14 days of Shuttle return, copies of all flight data acquired relevant to his experiment. Examples of such data include:

1. Records of the furnace hot zone (maximum) temperature and the temperature near the furnace wall ($\pm 30^{\circ}\text{C}$), if different, versus time, starting with power turn-on through experiment shut-down.
2. Accelerometer data during entire experiment run.
3. Time of specimen insertion into high heat zone of the furnace.
4. Record of specimen axial position in the furnace (± 1 cm) from immediately after specimen insertion into the high heat zone through activation of the retrieval device relative to the center of the high heat zone. Position versus time should be recorded.
5. Record of specimen radial position in the furnace (± 0.5 cm) from immediately after specimen insertion into the high heat zone through activation of the retrieval device versus time.
6. An indication of the specimen holding and insertion mechanism position after specimen insertion. (That is, has it retracted as planned?)
7. The time of cooling shroud insertion and an indication of its position after insertion. (That is, did it insert fully, or did it hang up at some intermediate position?)

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8. The time of retrieval device activation, and its position after activation.
9. Repeat steps 3-8 for each sample.
10. The time of furnace shutdown.
11. Pressure inside the canister (± 50 mm of Hg) versus time for the entire duration of the flight.
12. The undeveloped film from the motion picture camera (see next subsection).

7.3 MOTION PICTURE COVERAGE

The motion picture camera should be mounted in such a manner that the following data are recorded on one film strip:

1. Axial position of the specimen with respect to the furnace hot zone.
2. Motion of the specimen in the sonic well.
3. Approximate radial position of the specimen.
4. Time of incipient melting.
5. Time of complete melting (and assurance that complete melting did, indeed, occur).
6. Time intervals between Items 4 and 5 and between Item 5 and the attainment of maximum superheat temperature.
7. Time of insertion of the cooling shroud and its position after insertion.
8. Activation of the retrieval mechanism and its position after activation.

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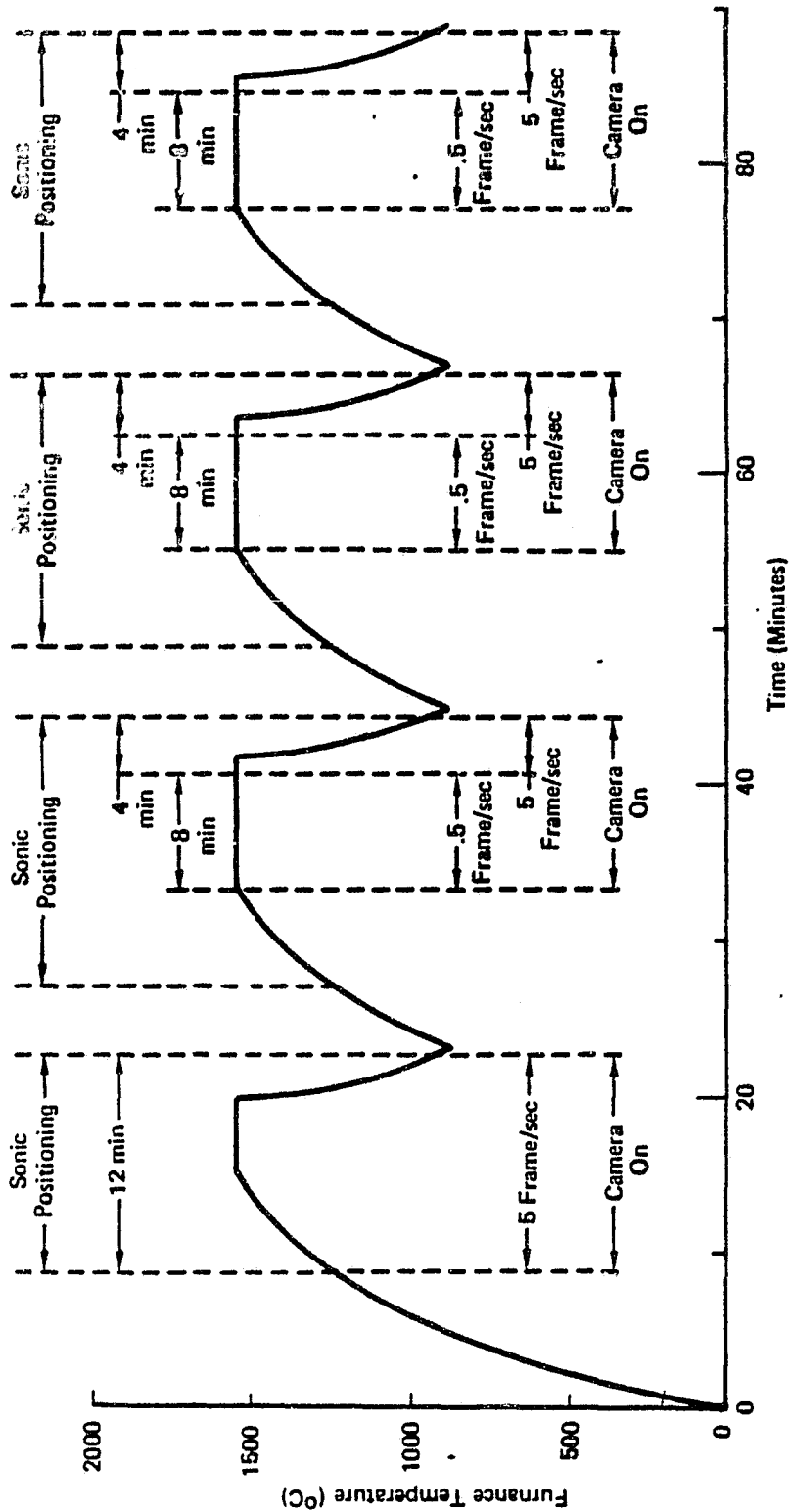


9. Appearance of the specimen versus time in the critical region between the solidus and superheat temperature. Since crystallization, if it should happen to occur, is an exothermic reaction, there is a significant brightening of the specimen at this time. If the specimen is observed when cooling, the time of crystallization could be determined.

In the event the specimen crystallizes, Items 5, 6, and 9 would be important for determining what to do differently in subsequent flights to increase the chances of glass formation.

The camera will be loaded with the same film (supplied by the PI) used for the characterization tests (see Subsection 5.3) and using the same filter as used for characterization photography. If it should prove desirable to obtain temperatures from the film (i.e., the returned flight specimen is obviously not 100% glassy), a second roll from the same lot of film could be exposed to laser melted oxides of known melting temperature by the PI using the flight camera, filter, and settings. The two rolls of film could then be developed under identical conditions in an automatic film processor. Densitometer readings of the ground-exposed film could be compared with those from the flight film to obtain critical flight temperatures of the specimen.

The camera presently planned to be used in the first MEA flight experiment has a film magazine carrying a maximum 200 ft long film roll. The limited length necessitates a careful planning of the use of the film. Taking advantage of the software capability to control the frame rate (i.e., number of frames per second), the following frame rate timeline, as shown in Figure 4,



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FIGURE 4. SPECIMEN FILMING TIMELINE

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is suggested:

1. The camera is turned on for Sample No. 1 (alumina sample) simultaneously as the acoustic levitator, with a frame rate of 5 frames/sec., and turned off (zero frame rate) simultaneously again as the levitator.
2. The camera is turned on for Sample No. 2 when it reaches 1600°C , with a frame rate of 0.5 frame/sec. The camera stays on for 8 minutes with this frame rate, at which time the rate increases to 5 frames/sec. The camera is turned off simultaneously as the acoustic levitator.
3. The filming operations for Samples Nos. 3 & 4 are identical to that for Sample No. 2 as far as the frame rates and the (relative) time of camera on/off are concerned.

It is to be noted that the above timeline for filming operations requires 7920 frames, which is within 1% of the total number of available frames (8000 frames = 200 ft), and therefore close following of the plan is mandatory.

7.4 ANALYSIS OF FLIGHT EQUIPMENT

The PI will be present to witness the opening of the flight canister. Following the opening, a detailed examination will be conducted under the direction of the PI. A NASA-provided still photographer will be available for color or black and white photography as needed. Examples of items that will be looked for and recorded photographically include:

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1. Location and condition of experiment specimen.
2. Location and quantity of condensed vapor inside the canister.
3. Location, quantity, and particle size of particulate matter originating from the canister contents.
4. Condition of the furnace, sonic generator, and other gear.
5. Condition of the flight battery and electrical connections.

It is anticipated that an Intersonics representative also will be available to assist in the examination and will have a list of items to look for.

7.5 ANALYSIS OF FLIGHT EXPERIMENT SPECIMENS

The PI will furnish appropriate packaging for the flight experiment specimens. After receiving the flight specimens, the PI will conduct a postflight analysis.

The analysis consists of the following tests/measurements:

1. Dimensions and weights
2. Bubbles
3. Surface crystals
4. Density
5. Internal stress
6. Homogeneity, striae
7. Refractive index
8. Absorption spectrum
9. Hardness
10. Devitrification temperature
11. X-Ray diffraction



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12. Spectrographic analysis

13. Wet chemical analysis

The alumina sample will undergo Test No. 1. For details of the test method/description/plan, the Sample Handling and Analysis Plan should be referred to. Following the analysis, a comprehensive report will be prepared describing the PI's activities and findings, starting with the beginning of the contract through the completion of the postflight analysis. Appropriate comparisons of MEA flight information with SPAR flight information will also be included.

8. MILESTONE SCHEDULE

Figure 5 shows a tentative schedule of activities and milestones. The schedule has been developed in reference to the experiment flight time. The post-flight analysis may involve some outside laboratories and therefore the indicated time is reserved to allow for individual lab test/reporting schedules.

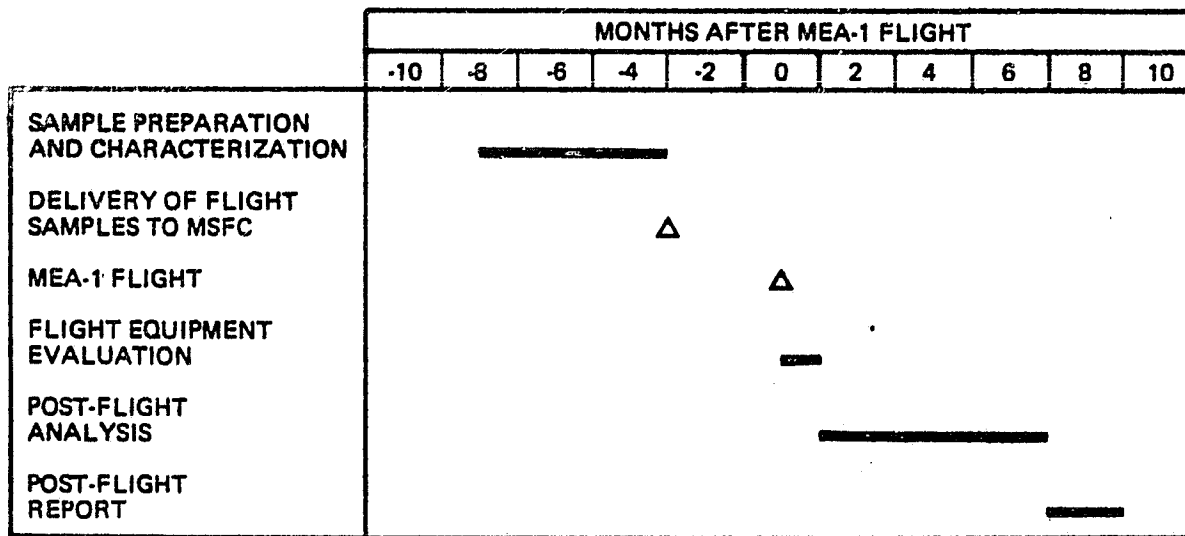


FIGURE 5. TENTATIVE SCHEDULE OF ACTIVITIES AND MILESTONES

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SSD81-0119

**SAMPLE HANDLING AND ANALYSIS PLAN
(SHAP)
FOR EXPERIMENT NO. 042
GLASS FORMATION-
MEA HIGH-TEMPERATURE SINGLE-AXIS
ACOUSTIC LEVITATOR (MISSION MEA-1)**

**NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
GEORGE C. MARSHALL SPACE FLIGHT CENTER
MARSHALL SPACE FLIGHT CENTER, ALABAMA**

JUNE 1981

PREPARED BY K.S. KIM

FOR

**PRINCIPAL INVESTIGATOR: R.A. HAPPE
(213) 594-3806/3940
CONTRACT NAS8-32953**



**Rockwell International
Space Operations and
Satellite Systems Division**

(NASA-CR-162073) CONTAINERLESS PREPARATION
OF ADVANCED OPTICAL GLASSES: EXPERIMENT
77F095 Final Report (Rockwell International
Corp.) 160 p HC A08/MF A01 CSCL 11B

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Unclas

SSD82-0125

CONTAINERLESS PREPARATION OF ADVANCED

OPTICAL GLASSES - FINAL REPORT

EXPERIMENT 77F095

March 1, 1982

Contract NAS8-32953

Prepared For
National Aeronautics and Space Administration
George C. Marshall Space Flight Center

Prepared By
R. A. Happe, Principal Investigator
And
K. S. Kim



Space Division
Rockwell International

SSD81-0119

**SAMPLE HANDLING AND ANALYSIS PLAN
(SHAP)
FOR EXPERIMENT NO. 042
GLASS FORMATION-
MEA HIGH-TEMPERATURE SINGLE-AXIS
ACOUSTIC LEVITATOR (MISSION MEA-1)**

**NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
GEORGE C. MARSHALL SPACE FLIGHT CENTER
MARSHALL SPACE FLIGHT CENTER, ALABAMA**

JUNE 1981

PREPARED BY K.S. KIM

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CONTRACT NAS8-32953**



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SSD81-0119

**SAMPLE HANDLING AND ANALYSIS PLAN
(SHAP)**

FOR EXPERIMENT NO. 042

**GLASS FORMATION -
MEA HIGH - TEMPERATURE SINGLE - AXIS
ACOUSTIC LEVITATOR (MISSION MEA-1)**

To

**NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama**

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For

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1. INTRODUCTION

This document describes a plan to characterize and evaluate both flight and flight experiment samples. The former refer to those that are to be flown and processed in space and the latter those that have been flown and returned to earth.

The flight samples will be analyzed mainly to determine their suitability for flight experiments and the flight experiment samples to verify the accomplishments of the experiment objectives and characterize these unique materials.

In the next section is given the background of the overall space glass experiment, which will be followed by sections on characterization process for flight and flight experiment samples, respectively. The sequence of the characterization process is given last.

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2. BACKGROUND

The combination of circumstances attending the melting of materials in space presents the possibility of making glasses from substances that, to date, have been observed only in the crystalline condition. A solid container is not needed during the melting and superheating portion of the manufacturing cycle. The only contact of the melt with its surroundings can therefore be a gaseous atmosphere or, if so desired, a vacuum. Thus, it is possible to melt many high-melting-point materials that heretofore could not be successfully melted because of reaction with the crucible material. This advantage alone might permit the preparation of new substances as glasses if their viscosity becomes sufficiently high on cooling to suppress crystal growth.

In addition, space melting permits cooling without the use of a solid mold. Thus many of the usual crystal nucleation sites are eliminated. Unless a given material can spontaneously nucleate on cooling, undercooling below the normal melting point will occur. If a sufficient amount of undercooling is accompanied by a sufficient increase in viscosity, crystallization will be avoided entirely and glass will result.

During the course of terrestrial research conducted by the PI over the past several years (NASA contracts NAS8-28014 and NAS8-28991), it has been shown that new glasses with potentially interesting optical properties can be prepared using a containerless melting technique. The technique most recently used for terrestrial research consists of suspending a molten drop of oxide material on a laminar flow air stream. The energy for melting is supplied by a 1 $\frac{1}{2}$ kW output CO₂ laser. The molten sample is steadied in the air stream

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by contacting it with a fused silica (glass) probe, or sting. The details of the techniques used and the results obtained are given in References 1 and 2.

The concept of glass making in space is of interest for the following reasons:

1. There is a strong possibility that oxides such as La_2O_3 , Ta_2O_5 , Nb_2O_5 , Al_2O_3 , Ga_2O_3 , and Y_2O_3 , and some of the rare earth oxides can be prepared as glasses in spherical boules of commercially useful sizes through space melting and cooling.
2. Glasses produced from such oxides or combinations of them with other oxide additions should have optical properties not obtainable in the conventional silicate-, borate-, and phosphate-based glasses.

It is envisioned that the process described need not be limited to oxide glasses. Perhaps other compounds, such as carbides, nitrides, and chalcogenides, will ultimately be producible as glasses by space processing.

If glasses could be prepared from oxides beyond the reach of current technology, the area of useful properties could be more than doubled, as is shown in the area labeled "potential space-produced glasses" in Figure 1. If the promise of space production can be realized by the preparation of simple compositions with properties falling roughly in the circles marked 1, 2, and 3, it should be possible, by combining the compositions, to fill in the areas shown in the figure. Thus, efforts to date have been largely devoted to attempts to prepare simple compositions with glass properties falling roughly within the three circles in Figure 1.

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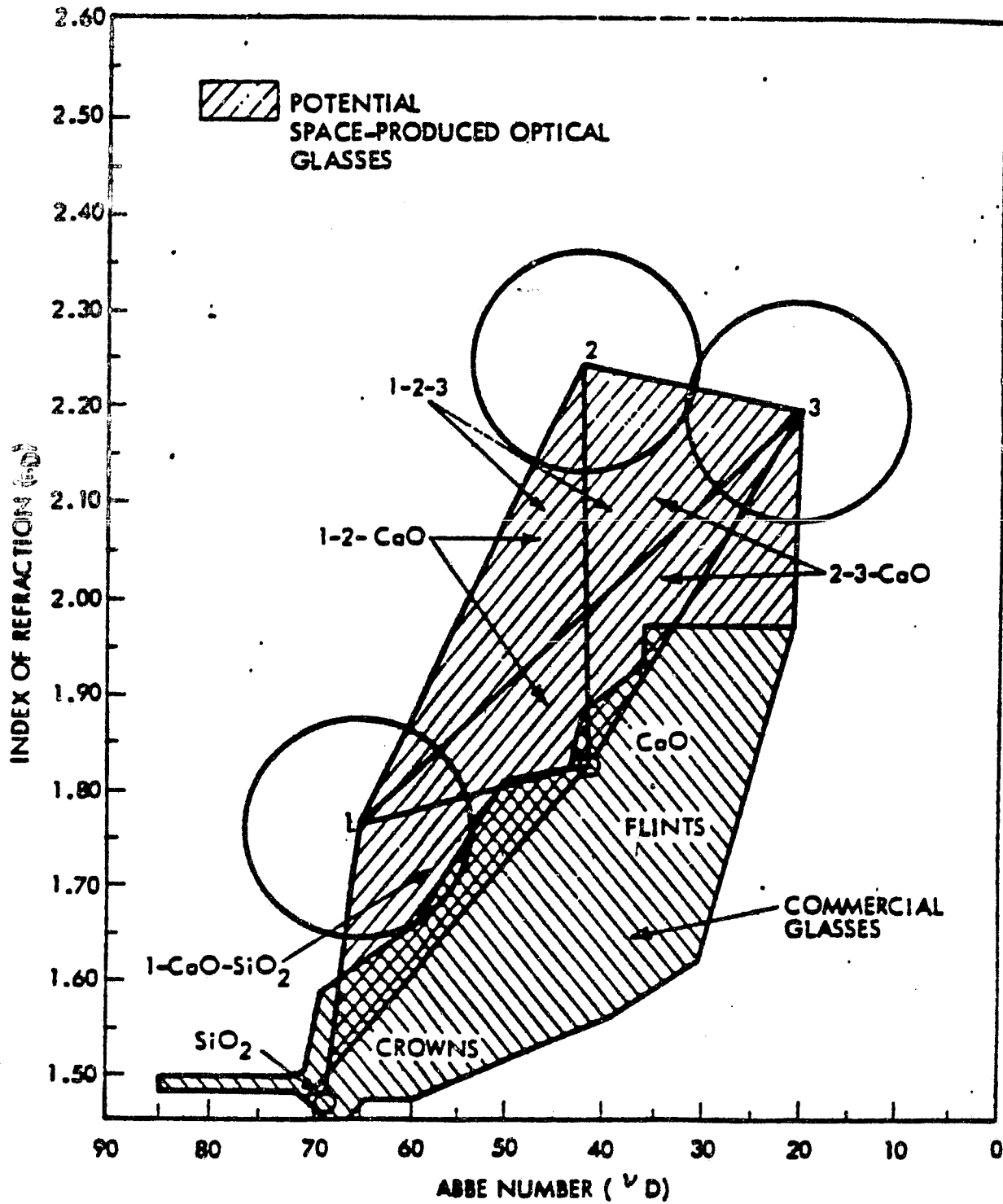


Figure 1. Potential Space-Produced Optical Glasses

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Additionally, there is another important area of application that new, space-produced, optical glasses might fill. Space production offers an opportunity to prepare new families of glasses free of the usual base oxides, SiO_2 , B_2O_3 , and P_2O_5 . Noncross-bred glasses should open possibilities for the lens designer for correcting "secondary spectrum." Reference 3 covers this subject in more detail.

3. CHARACTERIZATION PROCESS

The characterization process covers two types of samples, the flight samples and the flight experiment samples. The former refer to those prepared terrestrially for the purpose of flight experimentation and may come in the form of hot pressed, cast, or laser-prepared specimen. The latter refer to those prepared under the zero-gravity condition in orbital flights and presumably are of all glass structure.

Different samples may be handled differently according to their physical and chemical characteristics. Only through proper handling can the samples be preserved and their characteristics be maintained. In the particular case of the first MEA flight samples, past experience has shown that they will present no difficulty nor require unusual care as far as handling them is concerned.

The flight specimens will be analyzed mainly for properties that may significantly influence the results of the flight experiments. That is, tests on these samples will check the starting material requirements, determine the variables with which the flight experiments should be conducted, and determine also the proper procedure for handling the flight and the flight experiment samples. On the other hand, tests on the flight experiment samples will mainly verify the accomplishments of the flight experiment objectives, namely, production of unique glasses..



3.1 Flight Samples

In the first MEA flight experiment four samples have been scheduled to be investigated: a high melting point sample, two ternary samples (identical), and a binary sample. The high melting point sample is included in the group of the samples to be investigated for the purpose of studying the performance of the acoustic levitator. It has a melting point higher than the high temperature capability of the furnace and so it will remain solid during the entire furnace cycle. It has been chosen to be alumina. Levitation of this sample will provide information regarding the performance of the acoustic levitator in terms of levitating solid vs liquid (molten) material. The ternary samples are a silica-modified gallia-calcia and the binary a gallia-calcia composition. (See ERIP for the details of the compositions)

3.1.1 Handling Procedure

All three types of samples are known to be quite stable in normal room atmosphere. The silica-modified gallia-calcia and the gallia-calcia compositions have been proved, through experiments for the SPAR program, to be very stable in normal air for over a year. Alumina is well known to be stable.

Therefore no unusual care will be required in handling the samples. Storage in a clean, impact-mitigating environment, and avoidance of contact with bare hands are most notable features in the handling care.

Even though the samples are known to be impervious to moisture it is believed that the performance of the acoustic levitator is not. Therefore as a part of the effort to keep the levitator environment free of moisture the sample environment will be kept free of moisture as well so that mixing of the two will not result in a moist environment for the levitator. The sample packaging will contain a desiccant, and will be provided by the P.I.

3.1.2 Tests and Measurements

The following properties will be determined on representative examples of the ternary and binary flight samples. The alumina sample will be examined for Test No. 1.



1. Density, dimensions, weights

Measure these properties using standard tools and instruments.

2. Chemical analysis, if indicated on the basis of laser melting tests.

3. Impact resistance

Determine this by dropping the sample approximately 3 feet on a steel plate or concrete floor.

4. Resistance to deterioration when exposed to a nondesiccated air atmosphere

Check, if necessary, surface condition every 24 hours while exposing the sample to a normal room atmosphere. This will determine the nature of the proper atmosphere for sample preservation. As mentioned previously, the three types of samples for the first MEA flight are expected to be stable and do not require this test.

5. Devitrification temperatures

Since the flight samples are devitrified, the devitrification temperatures have to be known. This will be measured by heating the sample at a given temperature in the oven for 3-4 minutes and examining crystallization as indicated by opacity. The temperature will be raised in 5-10 degree steps.

6. Glass forming tendency

Terrestrial laser melting tests employing the technique of

air suspension with a silica probe will be performed on representative examples of the flight samples. The primary purposes of these tests are:

- i. To assure that the flight samples are capable of forming glass in the 0.6 cm ($\frac{1}{4}$ inch) size under containerless melting and cooling conditions.
- ii. To assure that the samples are free of gas formers that could cause excessive bubble formation during the flight experiment.
- iii. To determine whether the purity level achieved in the samples is sufficiently high to yield colorless glass.
- iv. To check the surface of the samples (compared with the interior) to determine whether a significant contamination exists.

7. Critical temperatures and times (cinematography study)

The method of determining critical temperatures was discussed in Section 6.3 of Reference 4. In all likelihood, the maximum temperature used for one of the SPAR flights will also be used for the first MEA flight.

3.2 Flight Experiment Samples

These are the samples that have been returned from the orbital flights. One of four samples is a solid with a high melting point, which is expected not to have undergone any significant change in its material characteristics, and therefore will not be extensively analyzed for

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...characterization purpose. On the other hand, the ternary and the binary compositions will have been melted and cooled containerlessly in space and are expected to have become glasses and hence warrant extensive analysis of their characteristics.

There will be three new glass spheres: two ternary and one binary. These will be the first space-produced glasses, and hence every effort will be made to preserve as many or as much of each sample as possible in their space-produced state. This will preserve the option for future examination of these space artifacts. It is expected that approximately a half of the binary and the ternary sphere, respectively, will be used in the post-flight analysis, and the remainder (one hemisphere of binary, one sphere and one hemisphere of the ternary) will be preserved.

3.2.1 Handling Procedure

The four flight experiment samples are expected to possess quite similar physical and chemical characteristics as the flight samples, and therefore the handling procedure for these will be the same as that for the flight samples. Desiccation of the sample environment inside the container is not necessary, unlike the flight samples.

3.2.2 Tests and Measurements

The following is a list of tests that will be conducted on the flight experiment samples. All the samples except the alumina sample will undergo Tests 1-5. The alumina sphere will be

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examined for Test No. 1. Tests 6-13 will be conducted with a half of the ternary and the binary sphere. The primary purposes of these tests are to determine if defect-free glasses of anticipated size have been obtained and to characterize the samples in terms of some basic characteristics.

AS SPACE-PRODUCED (SPHERICAL) SAMPLES

1. Dimensions and weights

These will be measured using standard tools.

2. Bubbles

These will be visually examined for using a microscope.

3. Surface crystals

SEM (scanning electron microscope) analysis will give qualitative compositions of surface crystals, the matrix, and the nucleator, if any.

4. Density

A standard technique will be employed.

5. Internal stress

This will be measured using a crossed-polarizer technique.

RECTANGULAR PRISMS PREPARED FROM SPHERICAL SAMPLES

(The samples will be stress-relieved prior to being cut into prisms)

6. Homogeneity, striae

Both will be visually examined. Inhomogeneity will manifest itself through areas of unequal refractive index, unequal

absorption characteristics, or unequal scattering characteristics, etc., and striae through areas of unequal refractive index. If the sample is homogeneous and free of striae its photographs would be featureless.

7. Refractive index

This will be measured at three wavelengths, and the dispersion will be calculated.

8. Absorption spectrum

The absorbance will be measured over a range of wavelengths, 0.33μ to 0.8μ . The measurements will be made on the same samples as used in Test 7.

9. Hardness

A standard tool will be used for this.

10. Devitrification temperature

The method for determining this is given in Section 3.1.2.

PULVERIZED SAMPLES

11. X-ray diffraction

The X-ray diffraction pattern will show the structure of the samples in terms of glassy or crystalline condition, and, if crystalline, the crystal type and the number of major phases present.

12. Spectrographic analysis

Pulverized samples will be placed between two electrodes and high voltages will be applied. The resulting sparks have characteristic patterns indicative of the major and minor

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elements present. A semi-quantitative analysis can be made from this.

13. Wet Chemical analysis

The chemical compositions are analyzed quantitatively through this analysis.

3.3 Sequence of Characterization Process

3.3.1 Flight Samples

All samples except alumina will undergo all the tests listed in Section 3.1.2 in the order presented. The alumina sample will be measured for Test No. 1.

3.3.2 Flight Experiment Samples

The tests listed in Section 3.2.2 will be conducted approximately in the order presented. However, since Tests 6-8 and Tests 9-13 may use different parts of a sample, they may be conducted concurrently.

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APPENDIX D

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The terrestrially prepared samples and the one prepared in the space environment will both be investigated for the properties listed in the following pages. The many measurements and tests required of the terrestrial samples are warranted by virtue of the uniqueness of the samples despite their non-space nature.

The list of the properties has been intended to be quite extensive in that it includes most of the characteristics pertinent to studying materials processing in space and/or to evaluating the applicability of such materials to practical needs. It was with the latter objective in mind, namely the less likelihood of application, that a number of material properties were omitted. Among them were many low (or cryogenic) temperature properties (especially low temp. expansion coefficient, thermal conductivity, heat capacity, bulk modulus, refractive index, absorption, magnetic susceptibility), magneto-optic constants, and piezo-electric effect.

The majority (over 20 items) of the tests are believed to be beyond the capability of principal investigator's organization and therefore subcontracting seems inevitable. Because of the large quantity and the specialized nature of the tests, some ten to twenty laboratories are expected to be involved, which may present a potentially overwhelming logistics problem let alone the cost.

It is proposed therefore, that a DRFP to measure all the above mentioned properties be sent to a number of laboratories encouraging them to respond at least to a part of the tests, and thereby the capability and the availability of the laboratories can be evaluated. The final list of the properties will be made after sufficient consideration is given to scheduling, the duration of the test, the cost, the type of the test (destructive/nondestructive, required size of the sample), etc.

DRAFT REQUEST FOR PROPOSAL TO CONDUCT
TEST/MEASUREMENTS ON SPECIAL GLASS MATERIALS

Rockwell International, under a contract from NASA, is carrying out a project to prepare special glass materials without a container. A glass sphere consisting of Ga_2O_3 and CaO will be prepared terrestrially using a wind tunnel and in the Space Shuttle. Both samples are expected to exhibit a number of unusual characteristics.

As is well known, the above composition does not form a glass when prepared terrestrially in a conventional manner: nucleation occurs from the container and rapid crystallization ensues because of its low melt viscosity. By eliminating the container and impurities, however, we have been able to prepare terrestrially a glass sphere of $\frac{1}{4}$ " diameter from this composition. Since this is the first time that the material has been prepared in a glassy state in $\frac{1}{4}$ " diameter size, it is eminently proper to fully characterize this unique material. This will help understand the science of glass formation and crystallization, and the physics of this material, and evaluate its usefulness.

It is expected that only one sample each will be available from the terrestrial and the Space Shuttle experiments. Measurements No. 1 to No. 3 will be done on a $\frac{1}{4}$ " (6.35mm) diameter sphere and measurements No. 4 to No. 30 on a cube of 2mm x 2mm x 2mm. To evaluate your capability and willingness to perform measurements and finalize the measurement plan, your response is requested regarding the following questions:

1. What measurements do you propose to perform?
2. Are the above sizes adequate for your measurements? If not, what is the minimum size?
3. If you cannot meet the temperature requirements, at what temperatures can you make measurements?
4. Will the sample maintain its original shape, size and internal structure after the measurements?
5. How long will it take to make measurements? How long to analyze the results?
6. How much will it cost?

1. Weight, Shape, and Dimensions

Measure/describe these for a spherical sample.

2. Cracks, Bubbles, Striae, and Surface Crystals (Rosettes)

Investigate for these using an optical microscope, etc., and if discovered, describe them as quantitatively as possible.

3. Internal Stress, Homogeneity, and Internal Structure (Gross)

By immersing the specimen in an appropriate liquid and using the crossed polarizer technique, investigate for internal stress and homogeneity. Determine also the gross internal structure in terms of glassy or crystalline state.

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4. Hardness

Measure this at room temperature using a standard hardness tester.

5. Optical Rotation, Dichroism, Birefringence, and Optical Anisotropy

The possession of any of these optical characteristics by the specimen can be checked in a straightforward manner using the crossed polarizer technique.

6. Chemical Composition and Corrosion-resistant Characteristics

Chemical composition will be analyzed in terms of percent distribution of Ga_2O_3 and CaO employing the wet chemical analysis technique. Corrosion resistance will be evaluated through a standard method.

7. Devitrification Temperature (Crystallization Temperature)

Determine this by melting the sample and letting the melt crystallize upon cooling.

8. Internal Structure (Detailed)

Using the powder X-ray diffraction method, determine the structure of the glassy state and that of the crystalline state. The atomic arrangements can be determined from the glass X-ray diffraction pattern, and the crystal structure from the crystal diffraction pattern.

9. Refractive Index and Dispersion (or Abbe Number)

The refractive index will be measured with the accuracy of 1×10^{-4} or better at three wavelengths (4861A, 5898A, and 6563A), and the Abbe number representing the dispersion will be calculated using the standard formula.

10. Absorption Spectrum

The absorption spectrum will be measured over a range of wavelength from infrared to ultraviolet not only to determine the absorption characteristics but also to measure the band gap.

11. Variation of Density (or Volume Expansion Coefficient)

The variation of the density will be measured as a function of time at an appropriate temperature or as a function of temperature with an appropriate heating rate, in order to determine the degree of departure of the density from its equilibrium value and thereby infer the cooling rate. The sample will be eventually heated to $1300^{\circ}C$ and then slowly cooled to room temperature. at which time the density will be measured. The sample will be slowly reheated to $1300^{\circ}C$ and this time highly quenched. The density will be again measured and compared with the previous value and that of the virgin sample. The density can be measured through the refractive index.

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12. Bulk Modulus

Measure the variation of the volume or the density as a function of mechanical stress at room temperature.

13. Effects of Pressure on Density

Measure the density (or refractive index) after a pressure is applied and removed noting the residual change in the density. Determine the pressure at which a significant residual change in the density occurs.

14. Viscosity

Measure the viscosity as a function of temperature between 300°C ~ 1300°C.

15. Heat Content (i.e., Change in Enthalpy)

Measure the change in enthalpy (at constant pressure) as a function of temperature between 300°C and 1300°C using the differential thermal analysis technique or by some other method. This will provide information on the gross internal structure and the cooling rate.

16. Heat Capacity (C_p or C_v)

Measure this at constant pressure or at constant volume at room temperature and as a function of temperature between 600°C and 1300°C.

17. Thermal Conductivity

Measure at room temperature and as a function of temperature between 600°C and 1300°C.

18. Infrared and Raman Spectra

Obtain these spectra using a broad range of infrared light to identify the defect groups and their transition modes.

19. Electro-optic Constants

Determine the Kerr and Pockels constants by applying high electric stresses (> 10KV/mm).

20. Stress-optic Constant (Photoelastic Constant or Brewster's Constant)

Determine this using a standard method.

21. Effects of Colorants

Select one, most interesting color center for this specimen, add it to the sample, and measure the absorption spectrum.

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22. Dielectric Loss Properties

Measure both the real and imaginary parts of the dielectric constant as a function of frequency (0 to 1 MHz or above) at room temperature and 200°C and two other temperatures between the two.

23. Dielectric Strength

Measure the dielectric breakdown strength (60Hz, ramp voltage) at room temperature.

24. DC Conductivity

Measure dc conductivity as a function of time (for up to 30 minutes) at selected temperatures (room temperature, 200°C, and two intermediate temperatures). DC conductivity will also be measured as a function of temperature for temperatures between 600°C and 1300°C.

25. Charge Storage Characteristics

Conduct TSC (thermally stimulated current) experiment and others, and determine the charge trap density, trap level, etc.

26. AC Conductivity

Measure ac conductivity as a function of frequency in the rf range.

27. Mechanical Loss Properties

Measure modulus (bulk, Young's or shear) as a function of frequency at frequencies from below 1 Hz to 1 MHz. Perform these experiments at room temperature, 200°C and two intermediate temperatures.

28. Mechanical Strength

Determine the mechanical strength (i.e., modulus of rupture) at room temperature.

29. Electron Paramagnetic Resonance and Nuclear Magnetic Resonance

Perform EPR and NMR experiments on the virgin and irradiated samples to determine the spin configurations of the host and the impurities (g values) before and after irradiation. Make the measurements with varying dose of gamma ray or fast neutrons.

30. Magnetic Susceptibility

Measure the magnetization or the magnetic induction as a function of magnetic field at room and a lower temperature.

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31. Cooling Rate and Devitrification Temperature*

Determine these for the flight sample by analyzing the temperature-sensitive flight photos.

* Applicable to the flight sample only.